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GROWTH OF GALLIUM ARSENIDE USING ION CLUSTER
BEAM TECHNOLOGY

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September 1988

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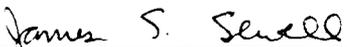
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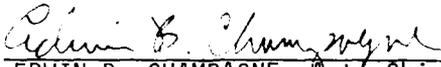
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This program was designed to study the feasibility of growing epitaxial GaAs thin films for subsequent application in electronic devices. Due to mechanical difficulties and design problems the technique of deposition via ionized clusters was not realized. Data collected is somewhat inconclusive. To answer the feasibility question further, work including modification of the hardware needs to be performed. To this point the techniques have been shown to be capable of growing single crystal GaAs, but the required electrical characteristics of the film are not present.

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PHASE II—FY 1984
PROJECT SUMMARY

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Growth of GaAs Using Ion Cluster Beam Technology

Technical Abstract (Unclassified) (Limit To Two Hundred Words)

This program was designed to study the feasibility of growing epitaxial GaAs thin films for subsequent application in electronic devices. Due to mechanical difficulties and design problems the technique of deposition via ionized clusters was not realized. Data collected is somewhat inconclusive. To answer the feasibility question, further, work including modification of the hardware needs to be performed. To this point the techniques have been shown to be capable of growing single crystal GaAs, but the required electrical characteristics of the film are not present.

Anticipated Benefits/Potential Commercial Applications of the Research or Development

A new method for the production of epitaxial GaAs layers for the fabrication of devices such as high-speed transmitters and receivers, lasers, light-emitting diodes, solar cells, detectors, etc.

EPI-TECH CORPORATION

FINAL REPORT

VOLUME I

TITLE : Growth of Gallium Arsenide using Ion Cluster Beam Technology

FOR : U.S. Air Force / AFWAL / XRPA AADR
Wright Patterson Air Force Base

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I.   INTRODUCTION

The following report is the culmination of three years effort whose
objective was to grow epitaxial gallium arsenide using "Ion Cluster
Beam" technology.

This effort included studies of process parameters such as ionizatiun,
acceleration, and substrate temperature, as well as the mechanical
parameters of source and nozzle design.

With the above objectives in mind it was felt that the hardware. i.e. -
the source and nozzle design plus the integrity of the process
environment in general, should be of high priority. This would insure
that process conditions would be repeatable and that any data gathered
would be reliable.

It is these ideas and concerns that have been the driving force behind
many of the hardware improvements and modifications.

II. BACKGROUND

A. ICB Technology

The ICB technology was developed at Kyoto University by Professor Takagi starting in the early 1970's. Initial studies have been directed at the deposition of thin, high quality metal films[1]. The films were of such materials as copper, silver, and gold, and were of very high quality. Even very thin films of gold, 100Å thick, were "pin-hole" free, had electrical resistivities near that of bulk gold, and had densities near theoretical values. Using conventional sputtering, e-beam or CVD techniques, such bulk properties of very thin films have not been obtained.

This technique has also been used in Japan to grow GaAs[2], GaP[3], GaN[4], and InSb[2]. The results on GaAs and InSb demonstrated single crystal x-ray patterns, but no electrical characterization was done. However, GaP was grown on GaP and compared to a commercially grown sample and showed similar results.

The standard ICB process, depicted in figure 1, is one of a class of techniques categorized as ion assisted film formation processes. Among other techniques in this same class are plasma deposition, magnetron sputtering, ion plating, and ion beam deposition.

The key to the ICB process is the formation of clusters, i.e. - an aggregate of atoms, at the exit of the source crucible.

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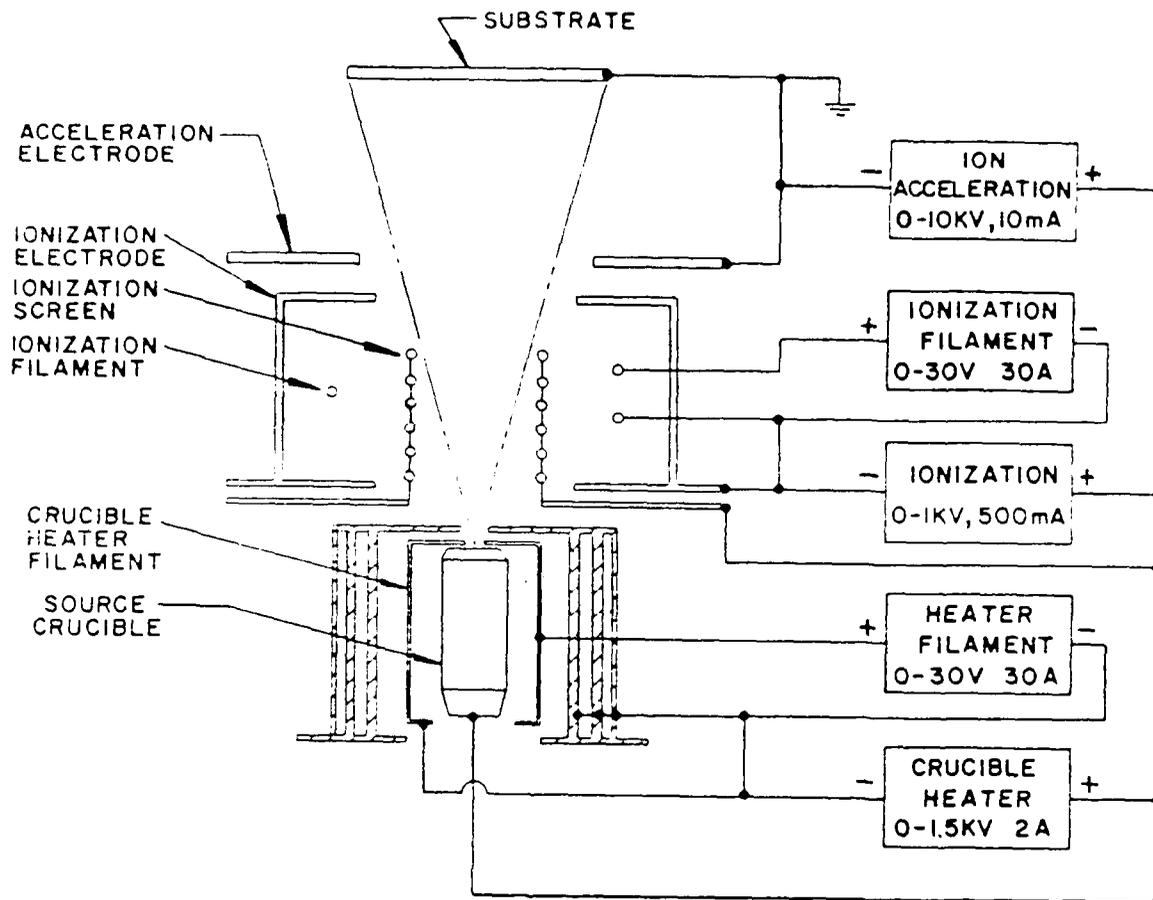
ION SOURCE ELECTRICAL SCHEMATIC

Figure 1

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For deposition of a metal or semiconductor, a vapor is ejected through a small nozzle at the top of the crucible. The temperature of the cell is maintained at some optimum value such that a pressure differential of several orders of magnitude is created across the nozzle. Under these conditions, rapid vapor expansion followed by supercondensation occurs to cause formation of aggregate clusters of between 500 and 2000 atoms.

The clusters can now be ionized, by variable intensity electron bombardment, in the ionization electrode assembly located above the crucible. Finally, the ionized clusters are accelerated toward the substrate by a variable potential applied to the accelerating electrode.

When the ionized and accelerated clusters plus the neutral clusters, at ejection velocity, arrive at the substrate surface, the impact causes the clusters to break up with an average kinetic energy per atom approximated by the following equations.

For neutral clusters:

$$\overline{E}_n = \tfrac{1}{2}mV_e^2 \,/\, N$$

where m = cluster mass
V_e = cluster velocity exiting the crucible
N = number of atoms/cluster

For charged clusters:

$$\overline{E}_c = \tfrac{1}{2}mV_e^2 \,/\, N \;+\; QV_a \,/\, N$$

where Q = charge on cluster
V_a = accelerating potential

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Note: These equations are very basic and neglect such items as thermal energy, electric field gradients, and cluster interactions.

By controlling V_a, it becomes possible to provide each atom in the ionized cluster with sufficient energy for enhanced surface diffusion, ($\overline{E}_c = 1$ eV), while at the same time maintaining the per atom energy low enough so as not to cause surface defects, ($\overline{E}_c < 5$ eV).

In addition, by controlling the percent of ionized clusters through the variable ionization potential, the total integrated beam intensity can be controlled.

With the ability to control these parameters it is in principle possible to control fundamental process effects such as sputtering, sticking, implantation, reactivity, and surface add-atom migration since these effects usually have a strong energy dependance[5]. Also, it should be possible to exercise some control over film characteristics like adhesion, structure and morphology.

Finally, this deposition process should provide the means by which films can be deposited at reduced substrate temperatures, since, upon impact of the clusters, a large portion of the kinetic energy of the cluster is translated into thermal energy based on the following relationship :

$$\overline{E}_c = [3/2 \ (KT)] \ / \ e \qquad \text{where } \overline{E}_c = \text{cluster kinetic}$$

$$\text{energy}$$
$$K = \text{Boltzmanns constant}$$
$$T = \text{Temperature}$$
$$e = \text{Electron charge}$$

While this is a simplistic representation of the actual process involved, it is sufficient to demonstrate that the surface temperature can be greatly enhanced through even small changes in the kinetic energy.

B. Phase I Review

1. History

The system used to grow GaAs for this phase of the project was developed by Eaton Corporation through collaborative efforts with Professor Takagi of Kyoto University.

The specific machine employed for deposition, is a multi-source machine using e-beams to heat each high purity graphite crucible. The graphite crucibles are shown in figure 2. The gallium crucible contained 15 grams of charge material and the arsenic crucible held from 5-7 grams of material.

The acceleration electrodes provide potential from 0-5 KeV on all sources simultaneously. Because of problems in the power supplies, the Phase I machine did not allow each source to be accelerated independently. Although the acceleration is a common value, the amount of ionization

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$$\overline{E}_c = [3/2 \ (KT)] \ / \ e$$

Nozzle Area

Graphite Cap

Graphite Crucible

Evaporant Material
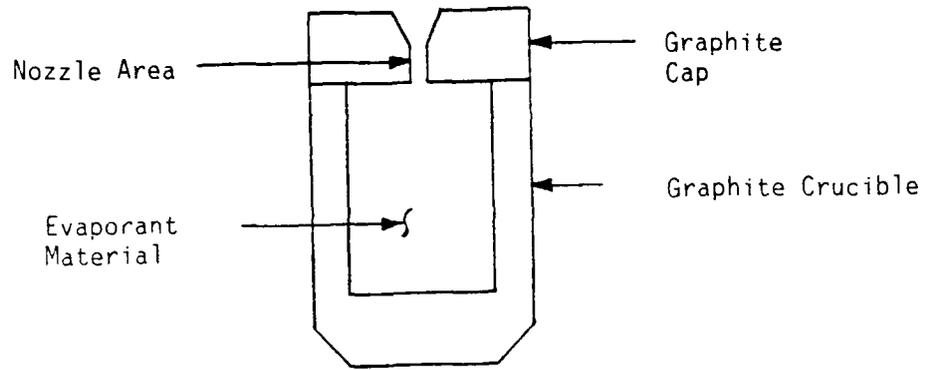
A. Crucible used in January

Nozzle Area

Graphite Cap

Evaporant Material

Graphite Crucible

Thermocouple
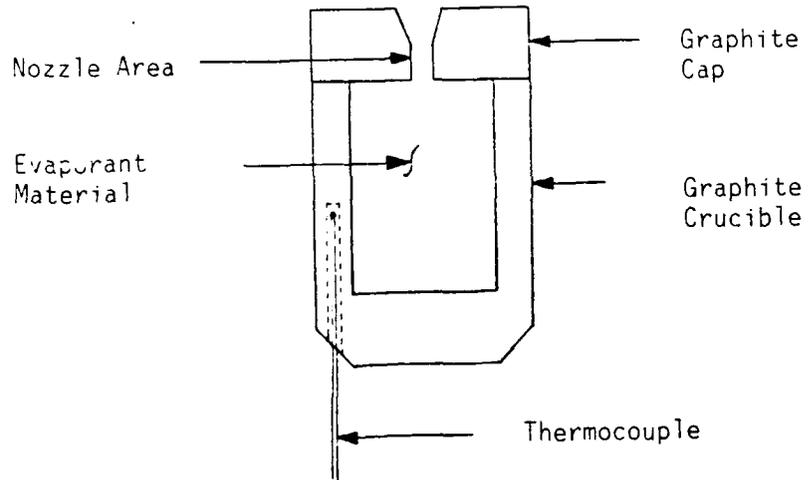
B. Crucible used in March

Figure 2

applied to each source as well as the power to the e-beam heater can be regulated separately.

The substrate area available for deposition is 10cm x 10cm and is heated by infrared lamps immediately above the sample. Substrate temperature is controlled by a thermocouple feedback system with the temperature able to be varied from room temperature to 800 °C and controlled very accurately by a Barber - Colman unit.

The sources are in a Balzers vacuum chamber with a large diffusion pump providing the system vacuum. The standard operating levels are 1-10 x 10^{-7} torr with no sample ionization and 1 x 10^{-6} torr with material deposition. On several occasions, the chamber door was opened to add arsenic after allowing sufficient time for cooling of the gallium source. Following recharge of the arsenic, the chamber was closed, and the pumping restarted. The typical cycle time from initial cool-down until resumption of growth was 2-3 hours. When growth was resumed, morphology, growth rates, and uniformity in thickness reproduced extremely well. Samples were loaded into the chamber through a turbo-pumped load lock by means of a walking beam mechanism. Total time for either loading or unloading was approximately 2 minutes. The deposition rate and thickness were monitored by a quartz crystal near the substrate using the appropriate calibration and tooling factors for the specific set-up and material.

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Although a Fluke controller is included on the system using a single crucible source, the multi-source system was operated purely in a manual mode.

The run procedure developed for the GaAs was different than that used for other ICB depositions on the same equipment because of the volatility of the arsenic from the wafer surface. In the first set of experiments conducted in January 1984, the wafers were etched in a $H_2SO_4O_2:H_2O$ (5:1:1) solution, rinsed in deionized water and blown dry with filtered nitrogen. The samples were then placed on a carrier shown in figure 3. Each of the two samples were 0.7" x 0.7" on edge and 0.014" thick.

Prior to loading each sample into the growth chamber, the gallium source was heated to 1225 °C, and the deposition rate was established by using the Inficon quartz crystal monitor system. After this was completed, the gallium beam was blocked by a manually operated shutter, and the arsenic source was then heated. Because of the low sublimation temperature of arsenic, only the heating elements of the e-beam heater were used without any electron bombardment to establish the deposition conditions. Because arsenic will not stick to the crystal monitor, the arsenic flux was monitored by watching the system pressure. This is a very difficult way to control the arsenic, since a change of 1 °C can alter the vapor pressure a significant amount.

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Figure 3

10
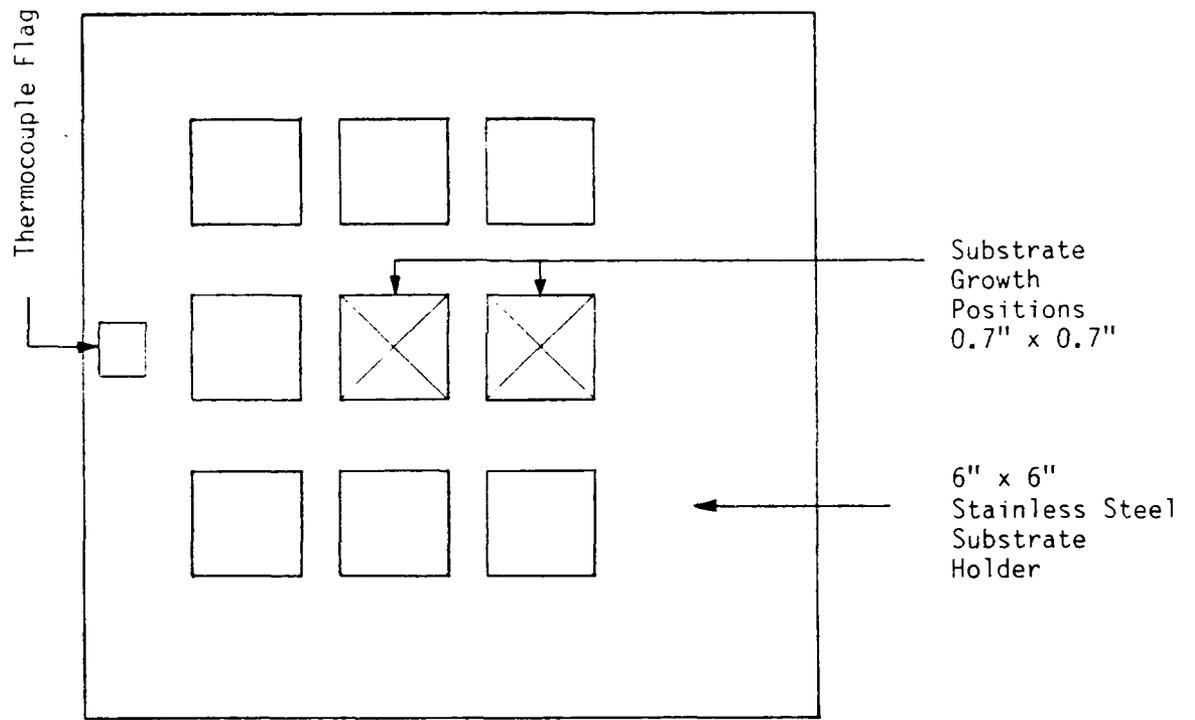
For some of the initial work, a thermocouple was placed in the side of the crucibles, as shown in figure 2B, and the temperatures read directly. This proved to be a problem, however, because the thermocouple was not floating at the same 10 KeV potential as the crucible, and thus caused arcing problems. Therefore, some runs, as noted on the data sheet, will show crucible temperature, but the majority will not.

Throughout a run, the operator was always trying to adjust the power supply to keep the pressure at a constant value. Once the arsenic was stabilized, the sample was moved into the growth chamber, the heating lamps turned on, and the shutter over the arsenic source opened to allow surface bombardment by the arsenic to prevent surface erosion due to arsenic evaporation. Once the sample was at temperature, the gallium shutter was opened, and growth initiated.

The ionization section on each source as well as the acceleration potential for some samples had been set prior to commencing growth. Although the machine is designed to have 10 KeV of acceleration, the presence of the arsenic vapor in the acceleration and ionization section causes arcing to occur, and thus limits the acceleration to 5 KeV. As stated earlier, the acceleration section was common for both the Ga and As source, so independent measurements could not be made. After growing the desired time, the gallium shutter was closed, and the heat was turned off. When the sample was

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below 400 °C, it was moved into the load lock and then into the atmosphere, and the cycle was repeated.

As the samples were evaluated following the first series of experiments, the sample thicknesses were very thin, i.e. - 100-500 Å.

The lack of growth precipitated some procedure and hardware changes for the second set of experiments done in March 1984. For the hardware, the change involved the redesign of the heater around the gallium crucible so that more power could be applied to the top of the crucible. In this manner, the "spitting" seen in the first set of runs with 6% power was absent in the second set at 16%. As can be seen in figure 4, the longer heater kept the nozzle area hotter, thus reducing condensation in this area.

Three significant changes were made in the deposition procedures for the second set of runs. The first involved the control of the arsenic pressure. The RGA was used to monitor the arsenic partial pressure in the chamber with manual controls still being used to adjust the temperature to compensate for changes. This procedure was more sensitive in controlling the arsenic pressure than using only the chamber pressure gauge.

The second change involved wafer clean-up. After struggling with inadequate wafer clean-up stations, it was decided to

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Nozzle Area

Evaporant
Material

Crucible Heater

Graphite Cap

Graphite Crucible

Heater Filament
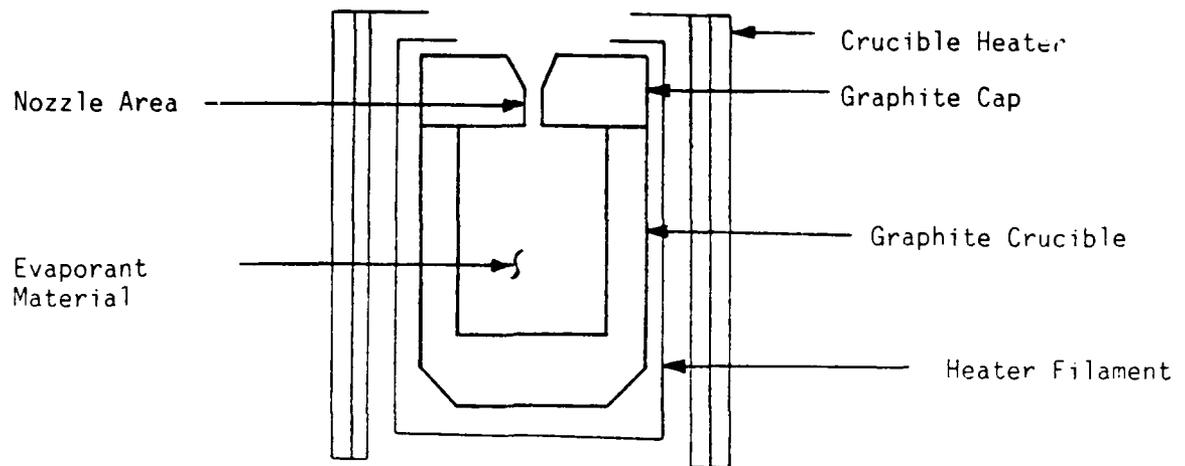
A. Original Configuration

Nozzle Area

Evaporant
Material

Crucible Heater

Graphite Cap

Graphite Crucible

Heater Filament
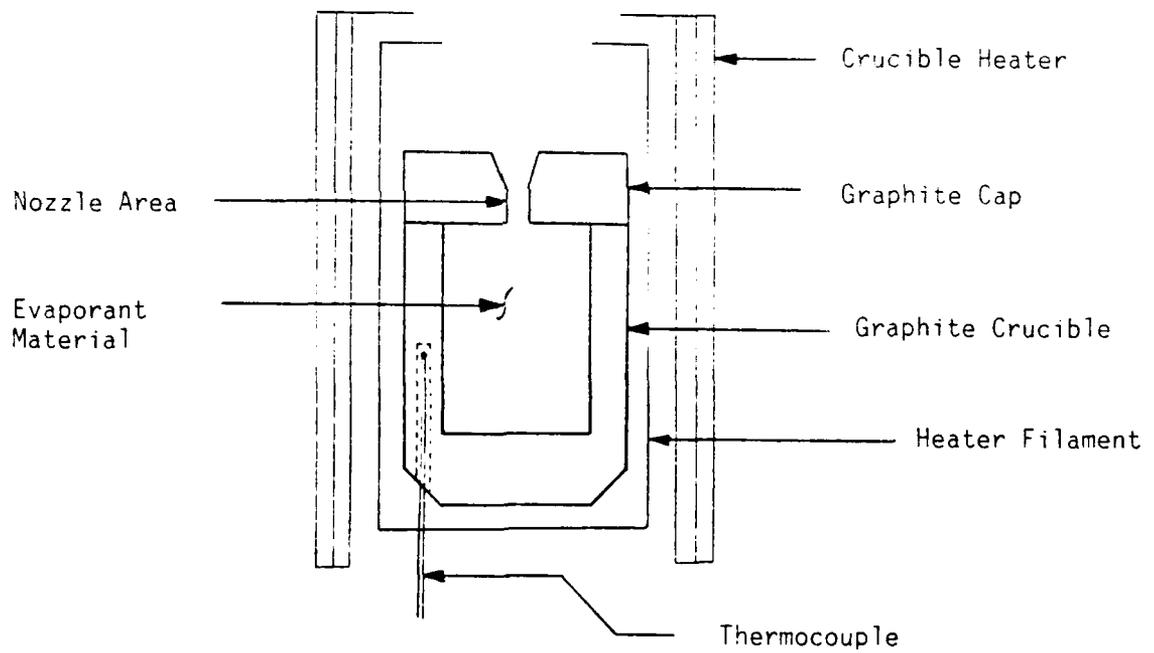
Thermocouple

B. Modified Configuration
(Lengthened Heater and Thermocouple)

Figure 4

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grow on the substrates with no initial clean-up either by solvent or acid treatment. The surfaces of the grown wafers were excellent as can be seen in the Phase I results section of this report. This ability to grow on a substrate with no clean up prior to deposition is truly unique to this deposition technique.

The third significant change involved the heating of the substrate. In the first set of experiments, the lack of substantial growth could have been attributed to the very low substrate temperatures. The low temperatures were due to the infrared light transmission characteristics through the GaAs as opposed to the absorption of infrared by metals or other semiconductors such as silicon. In order to get better absorption, three different approaches were tried. In one approach, a piece of stainless steel was placed over the sample with no bonding material. A second approach had various thicknesses of gallium deposited on the back of the wafers. The third approach had a thermal bonding agent, either gallium or indium, on the back of the wafer holding it to the stainless steel back plate. This latter approach was most successful, although it required the most time for preparation and de-mounting. A schematic can be seen for the three methods in figure 5.

Using the modified hardware and process parameters, films were successfully grown using the ICB equipment. However,

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○ ○ ○ ○ ○ ○ ○ ○ ○ ○ ○ ← Heater Lamps

GaAs Substrate

Stainless Steel
Holder

A. Substrate Only

Stainless Steel
Backing

GaAs Substrate

Stainless Steel
Holder

B. Substrate with Unbonded Backing

Gallium Metal
Film 3000-8000 Å

GaAs Substrate

Stainless Steel
Holder

C. Substrate with Gallium Film

Gallium or Indium
Metal

Stainless Steel
Backing

Stainless Steel
Holder

GaAs Substrate

D. Substrate with Thermal Bonding Agent and Backing

Figure 5

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the equipment had several problems that still needed to be corrected for future work with GaAs.

First, the heaters for both Ga and As needed to be altered so that very precise temperature control can be maintained on both sources, with each source being uniformly heated.

A second major problem involved the power supplied within the system. Because the sample was at 0 potential, all the power supplies operate on top of a 10 KeV potential which causes many problems. As a result of these conditions, modified power supply designs were studied and implemented. Also, a machine to do GaAs would need an RGA that will go to 300 a.m.u. and a quad head that could be used for some flux measurements in the deposition area. Such hardware modifications and additions were evaluated and included in the next generation GaAs system. By making these and other small changes, the redesigned hardware would support the process for growth of GaAs in a more controlled and reproducible manner.

2.   Run Parameters Summary

The run conditions will be divided into two groups. Table I will represent the work done in the January experiments, and Table II will represent the results from the March work.

As can be seen in both tables, pieces of data on specific run conditions are omitted. In most cases such as the

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temperature readings for the Ga and As crucible. no read-outs were available in January. In March. the high voltage in the system interacted with the thermocouple on many occasions. causing arcing or blowing power supplies. As discussed in an earlier section. only the heater filament of the arsenic crucible was turned on because of the low temperature requirements for arsenic. In Table I. thickness measurements are not stated for all samples because of the very thin layers. The cleaning process and mounting procedures made it impossible to use a surface profileometer such as a Dek-Tak to measure a step and the standard stain and cleave procedures will not resolve layers <3000 Angstroms. The deposition rate numbers were found to be very erratic throughout the program, thereby making these values suspect. In some of the last runs. correction factors were included to help modify the numbers and to correlate with the values measured by Dek-Tak. When the corrections were made. deposition rates of 2.4-2.6 $\overset{\circ}{A}$/sec were observed. giving growth rates of 120-150 $\overset{\circ}{A}$/minute. The geometry of the system will make the crystal monitor very inaccurate until modifications are made. Also. the acceleration values for Ga and As are not always recorded as being equal. In reality. they are equal because of the hardware wiring. The discrepancy is merely from the set point of the arsenic not being adjusted to read the same as the Ga.

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DEFINITION OF PARAMETERS

Substrate   This indicates the doping of the GaAs - Si=Silicon   U=Undoped   Cr=Chromium

FV (V)   Forward voltage in volts of filament on Ga crucible
FI (V)   Forward current in amps of filament on Ga crucible
SV (V)   e-beam supply voltage in volts on Ga crucible
SI (mA)   e-beam supply current in mA on Ga crucible

FV (V)   Forward voltage in volts of Ionization section above Ga crucible
FI (V)   Forward current in amps of Ionization section above Ga crucible
SV (V)   Supply voltage of e-beam source in Ionization section above Ga crucible
SI (mA)   Supply current of e-beam source in Ionization section above Ga crucible
Accel KeV   Acceleration potential on Ga beam in KeV

FV (V)   Forward voltage in volts of filament on As source
FI (A)   Forward current in amps of filament on As source
SV (V)   e-beam supply voltage in volts of filament on As source
SI (mA)   e-beam supply current in mA of filament on As source

FV (V)   Forward voltage in volts of Ionization section above As crucible
FI (A)   Forward current in amps of Ionization section above As crucible
SV (V)   Supply voltage of e-beam source in Ionization section above As crucible
SI (mA)   Supply current of e-beam source in Ionization section above As crucible
Accel KeV   Acceleration potential on As beam in KeV

Pressure   Pressure in chamber in Pascal
Ga crucible temp.   Temperature of Ga crucible
As crucible temp.   Temperature of As crucible
Substrate temp.   Indicated temperature of substrate by thermocouple monitor
Deposition rate   Deposition rate as read on crystal monitor - A/sec
PWR   Power on Ga crucible as a % of total available power
Thickness   Thickness of epi layer grown

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TABLE I

Run #	1	2	3	4	5	6	7	8	9	10	11	12
Substrate	Si	Si	Si	Si	Si	Si	Cr	Cr	Cr	Cr	Cr	Cr
Ga Crucible FV	42	41.6	42.2	42.1	41.9	42.8	41.5		40.2	40.4	40.9	39.7
Ga Crucible FI	17.1	17.0	17.0	17.1	17.2	17.0	17.0		16.7	16.8	16.9	16.7
Ga Crucible SV	360	360	360	350	400	400	350		360	350	340	380
Ga Crucible SI	520	530	540	540	460	580	460		430	460	470	410
Ga Ionization FV	43.9	12.2	11.0	11.3	11.5	11.0	12.3		12.4	12.2	12.1	12.2
Ga Ionization FI	17.3	11.1	10.5	10.7	10.7	10.4	11.2		11.2	11.2	11.2	11.2
Ga Ionization SV	---	300	100	100	100	110	300		300	290	300	300
Ga Ionization SI	---	337	113	133	128	92	315		308	335	285	269
Ga Accel.	0	0	1	2	4.1	4	3		3	4	5	3
As Crucible FV	5.7	5.4	5.0	5.0	5.9	5.7	5.8		5.9	5.0	5.3	5.3
As Crucible FI	5.3	4.8	4.7	4.7	4.9	4.7	4.7		5.1	4.5	4.6	4.6
As Crucible SV	---	---	---	---	---	---	---		---	---	---	---
As Crucible SI	---	---	---	---	---	---	---		---	---	---	---
As Ionization FV	---	13.2	11.9	12.6	---	13.5	13.7		13.2	13.5	13.6	13.7
As Ionization FI	---	11.4	10.9	11.4	---	12.3	12.4		12.0	12.2	12.4	12.4
As Ionization SV	---	300	110	110	---	100	310		300	310	310	310
As Ionization SI	---	211	---	158	---	253	305		288	332	276	285
As Accel.	0	0	1	1	4	4	3		---	4	---	3
Pressure	$3X10^{-4}$	$5X10^{-4}$	$3X10^{-4}$	$5X10^{-4}$	$3X10^{-4}$	$4.5X10^{-4}$	$7X10^{-4}$		$6X10^{-4}$	$5X10^{-4}$	?	$5X10^{-4}$
Ga Cruc. temp.	---	---	---	---	---	---	---		---	---	---	---
As Cruc. temp	---	---	365	365	---	---	---		---	---	---	---
Sub Temp.	601	600	600	600	600	600	600		600	550	500	600
Dep'n. Rate	3.4	---	---	---	2.5	3.2	3		4.2	4.6	5.8	3.9
Power (%)	7	7	7	7	7	7	6		7	7	6	6
Thickness	0	0	0	?	<3000	<3000	<3000		<5000	?	?	?

Run 8: Ran out of As before run conditions were established and recorded

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TABLE II

Run #	1	2	3	4	5	6	7	8	9	10	11	12
Substrate		Cr	Cr	Cr	U	U	[a]	U	U	U	U	[b]
Ga Crucible FV		30.0	30.3	30.3	30.2	30.2		29.9	29.6	29.7	31.2	
Ga Crucible FI		16.3	16.3	16.3	16.4	16.4		16.0	16.2	16.3	16.8	
Ga Crucible SV		790	700	690	790	790		670	860	890	780	
Ga Crucible SI		520	690	660	590	690		690	560	520	590	
Ga Ionization FV		12.7	12.7	12.5	13.1	13.1		12.4	12.4	12.5	12.7	
Ga Ionization FI		11.6	11.6	11.6	11.8	11.8		11.4	11.4	11.5	11.6	
Ga Ionization SV		300	310	320	299	300		360	300	300	300	
Ga Ionization SI		299	374	267	340	340		285	278	269	275	
Ga Accel.		3.0	3.0	3.0	3.0	3.0		3.0	3.0	3.0	3.1	
As Crucible FV		6.1	5.7	5.9	4.8	4.8		6.0	5.0	5.2	4.9	
As Crucible FI		5.1	5.0	5.0	5.3	5.3		6.0	5.4	5.5	5.3	
As Crucible SV		--	--	--	--	--		--	--	--	--	
As Crucible SI		--	--	--	--	--		--	--	--	--	
As Ionization FV		12.3	--	--	11.5	11.5		12.1	12.1	12.1	12.6	
As Ionization FI		10.6	--	--	9.8	9.8		10.2	10.3	10.3	10.4	
As Ionization SV		--	--	--	90	300		300	300	300	300	
As Ionization SI		--	--	--	61	011		11	12	15	13	
As Accel.		--	--	--	--	--		--	--	--	--	
Pressure		8X10⁻⁴	8X10⁻⁴	7X10⁻⁴	7X10⁻⁴	7X10⁻⁴		4X10⁻⁵	8X10⁻⁴	8X10⁻⁴	10X10⁻⁴	
Ga Cruc. temp.		--	1225	1225	1225	1225		--	1220	1220	--	
As Cruc. tcmp		368	--	371	325	325		340	344	353	--	
Sub Temp.		600	600	600	600	600		600	600	600	--	
Dep'n. Rate		6.0	6.5	5.5	--	--		--	--	--	--	
Power (%)		16	16	16	16	16		16	17	17	16	
Thickness		3500	?	?	2100	3000		2800	4000	14000	?	

Note [a] (Run 7): Ga deposition backs of wafers only. No As. Done to check absorption properties.

Note [b] (Run 12): Run aborted -- Mechanical Problem loading sample into chamber

(Pressure exponents as printed: 8×10^{-4}, 7×10^{-4}, 4×10^{-5}, 10×10^{-4}.)

Table II (cont.)

Run #		13	14	15	16	17	18	19	20	21	22	23	24
Substrate		U	U	U	U	U	U	U	U	U	U	U	U
Ga Crucible	FV	31.4	30.6	30.6	30.6	33.0	30.4	31.0	31.7	31.1	30.5	30.3	31.4
	FI	16.8	16.6	16.6	16.6	18.1	16.3	10.5	16.7	16.6	16.4	16.3	16.4
	SV	730	730	730	730	720	730	720	710	720	700	730	560
	SI	650	630	630	630	450	650	780	640	620	700	620	830
Ga Ionization	FV	12.5	12.6	12.6	12.6	10.9	---	12.7	12.6	13.0	13.0	12.6	12.7
	FI	11.5	11.6	11.6	11.6	10.5	---	11.6	11.6	11.7	11.7	11.6	11.6
	SV	310	310	310	310	290	---	300	310	300	300	310	300
	SI	301	310	310	310	278	---	233	?	300	300	300	291
Ga Accel.		3.0	3.0	3.0	4.0	---	---	3.0	3.0	1.0	2.0	4.6	0
As Crucible	FV	5.2	4.0	4.0	4.0	6.6	6.6	6.5	5.4	5.9	6.4	5.9	6.2
	FI	6.6	5.1	5.1	5.1	5.3	5.4	5.0	5.5	4.6	4.8	4.5	4.6
	SV	---	---	---	---	---	---	---	---	---	---	---	---
	SI	---	---	---	---	---	---	---	---	---	---	---	---
As Ionization	FV	12.0	12.4	12.4	12.4	10.9	---	12.2	12.1	12.2	12.1	12.0	12.1
	FI	10.3	10.5	10.5	10.5	9.6	---	10.4	10.2	10.2	10.2	10.2	10.3
	SV	310	290	290	290	290	---	300	300	300	300	310	310
	SI	13	20	20	20	14	---	11	11	11	11	12	15
As Accel.		3.0	3.0	3.0	4.0	0	---	3.0	3.0	1.0	2.0	4.6	0
Pressure		9×10^{-4}	9×10^{-4}	9×10^{-4}	9×10^{-4}	9×10^{-4}	9×10^{-4}	9×10^{-4}	7×10^{-4}	7×10^{-4}	7×10^{-4}	7×10^{-4}	7×10^{-4}
Ga Cruc. temp.		1210	1210	1216	1216	---	---	---	1220	1220	1220	1220	---
As Cruc. temp		---	---	---	---	---	---	---	---	---	---	---	---
Sub Temp.		500	400	300	400	400	400	400	500	400	400	400	300
Dep'n. Rate		---	---	---	---	---	---	---	---	5.0	4.5	2.4	2.4
Power (%)		16	16	16	16	16	16	16	16	16	16	16	16
Thickness		10,000	---	10,000	5,000	6,000	0	20,000	50,000	10,000	8,500	9,000	8,500

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Table II (cont.)

Run #	25	26	27	28	29	30	31	32	33	34	35
Substrate	U	U	U	U	U	U	U	U	U	U	U
Ga Crucible FV	31.7	32.1	31.2	31.3	30.9	31.9	29.2	32	32	31	31
Ga Crucible FI	16.6	16.8	16.5	16.4	11.5	16.6	15.8	16	16	16	16
Ga Crucible SV	550	670	610	600	650	560	810	650	470	560	550
Ga Crucible SI	900	720	780	810	720	830	550	750	990	850	800
Ga Ionization FV	12.7	12.9	13.0	12.7	12.6	12.7	12.6	13.0	13.0	9.9	9.6
Ga Ionization FI	11.5	11.7	11.7	11.6	11.6	11.6	11.5	11.0	11.8	9.8	9.9
Ga Ionization SV	310	310	300	300	300	310	310	300	300	310	320
Ga Ionization SI	300	300	300	300	300	310	309	300	389	7	10
Ga Accel.	1	2	4	3	4.8	3.0	2.7	3.0	2.5	3.0	3.0
As Crucible FV	6.9	6.5	6.0	5.8	6.0	5.8	6.4	7.3	9.1	7.2	7.3
As Crucible FI	5.0	4.9	4.4	4.5	4.6	4.5	5.0	5.1	6.1	6.4	6.4
As Crucible SV	---	---	---	---	---	---	---	---	---	---	---
As Crucible SI	---	---	---	---	---	---	---	---	---	---	---
As Ionization FV	11.8	12.7	12.2	12.2	12.4	12.2	11.9	?	?	12.3	12.9
As Ionization FI	10.1	10.3	10.1	10.2	10.3	10.2	10.0	?	?	10.3	10.7
As Ionization SV	310	300	300	300	300	300	300	?	?	300	290
As Ionization SI	10	11	10	11	11	11	9	?	?	14	32
As Accel.	1	2	4	3	4.8	3	2.7	3	?	3	3
Pressure	8×10^{-4}	7×10^{-4}	7×10^{-4}	7×10^{-4}	6×10^{-4}	7×10^{-4}	6×10^{-4}	9×10^{-4}	1×10^{-3}	1×10^{-3}	1×10^{-3}
Ga Cruc. temp.	---	1225	1225	1225	1225	1207	1165	---	---	1230	1225
As Cruc. temp	---	---	---	---	---	---	---	---	---	---	---
Sub Temp.	300	300	300	400	400	500	500	400	400	400	400
Dep'n. Rate	2.4	1.7	2.2	---	---	2.2	5.5	---	---	---	---
Power (%)	16	16	16	16	16	16	16	16	16	16	16
Thickness	5000	12,500	10,000	5,000	12,500	21,300	?	7,000	?	?	?

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3. Experimental Results

In the Phase I proposal submitted and awarded, the
anticipated results were:

a. Growth of single crystal GaAs on GaAs substrates
    with Ga and arsine.

b. Parameters for growth of GaAs epi on GaAs
    substrates at temperatures below 600 °C.

c. Characterization of the films as to their
    electrical and physical properties.

Of these anticipated objectives, only one was modified. A
single source machine had been expected at the time the
proposal was written, and it would have necessitated the use
of Ga and AsH_3 as the starting materials. The availability
of the multi-source machine in time for the work made this
approach unnecessary since now both metallic gallium and
metallic arsenic could be used. With the exception of this
one change, all the objectives of the proposal were met.

This section will discuss in detail the experimental methods
used and the results obtained by each technique in the
characterization of the electrical, chemical, and physical
properties of the samples grown.

a. Film Thickness Uniformity and Morphology

The thickness of the films was measured by two
techniques: surface profiling and cleaved cross

23

sections. The surface profiling was done using a Dek-Tak system sold by Sloan Instruments. In this simple test, a step height was measured from the grown film to the starting substrate. As can be seen in figure 6 the holder provides a "masked" area for the substrate. This method worked exceedingly well in the March work where samples were not etched in the $H_2SO_4:H_2O_2:H_2O$ (5:1:1) mixture prior to deposition. This etch "rounded" the surfaces at the wafer edges and made the Dek-Tak ineffective. With the second set, well defined traces were obtained as shown in figure 6 with clearly defined steps of 4000 Å and less.

The second technique used was the conventional cleave and stain technique. A selective etch, $CrO_3:HF:AgNO_3:H_2O$, was used to delineate the film/substrate interface. Using a high power optical microscope equipped with differential interface contrast (DIC), the thickness could easily be measured. This technique, however, is limited to films with thicknesses of > 3000 Å. Using this technique, the substrate/epi interface can be evaluated for smoothness. A given sample was cleaved into four pieces, and the thickness was measured at 12 points across the sample. The values were all within ±10% at each location. This ±10% is the experimental error of the technique. On the sample measured, the thickness was 1.0±0.1μm for the 0.7" x

24
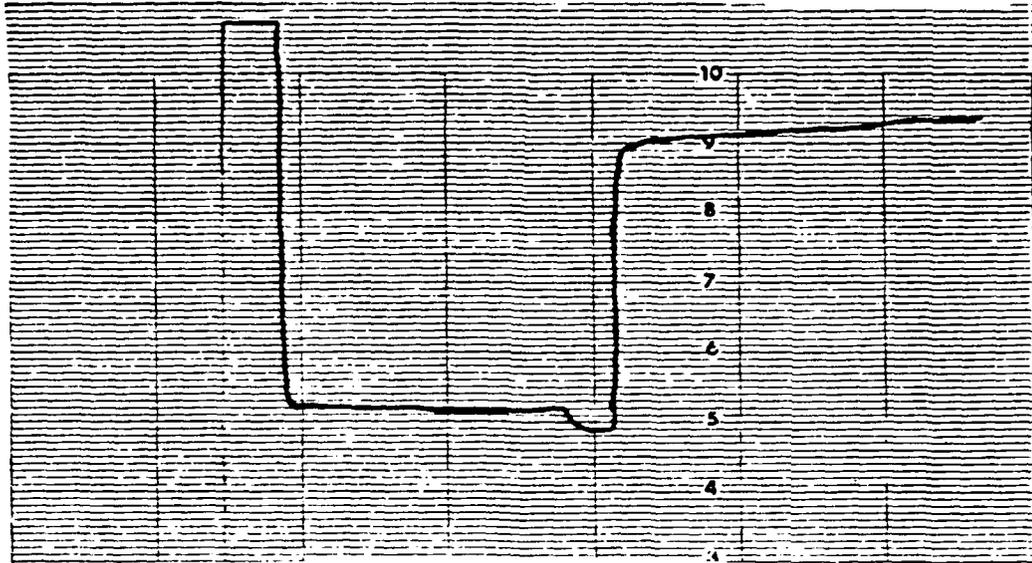
A.   Sample Number 9,   1000 Å per division,   approximate thickness=3900 Å
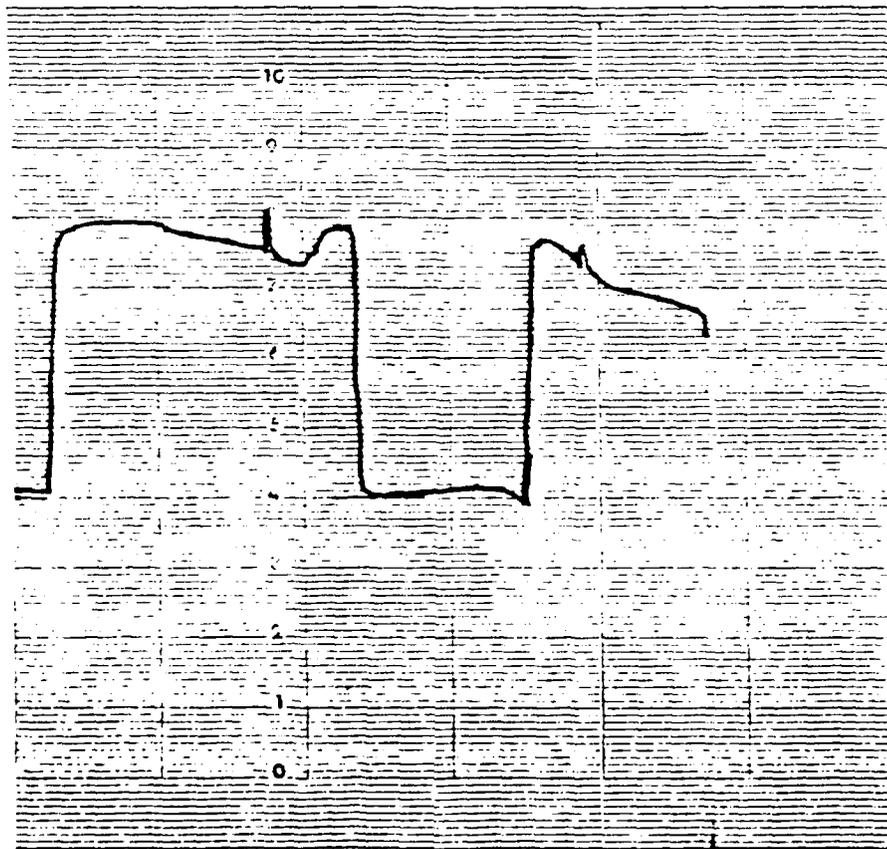
B.   Sample Number 19,   5000 Å per division,   approximate thickness =
     2 microns,   this was the first 2 inch wafer grown.

Figure 6

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0.7" sample. The Dek-Tak value was 9000 Angstroms for the same sample. Three two-inch diameter samples were also grown, but the best sample was used for electrical data, and the other two samples had mechanical problems that caused non-uniform thicknesses. In one sample, a shutter partially shielded the sample, and in the second, the arsenic was depleted, and the run continued longer that it should have. On the only good sample, the perimeter values appeared very uniform around the wafer as determined by Dek-Tak, but the center values were not measured by the cleave and stain method. The uniformity was checked on a second sample and was found to have the same range of values, i.e. - $\pm 10\%$.

On all samples evaluated by the cleave and stain method using DIC, the interface was very smooth. The smooth interface on the samples which had not previously been cleaned, is indicative of a unique process. Such interfaces on either a VPE or MBE sample with no cleaning would not have the same high quality.

Further evidence of the unique film deposition parameters is the surface morphology. As can be seen in the micrograph of the surface in figure 7, the surface of both the substrate and epitaxial film are identical. This epitaxial film is 1.0μm thick and appears the same as the uncleaned substrate area when both are examined

Epi Layer

Transition

Substrate
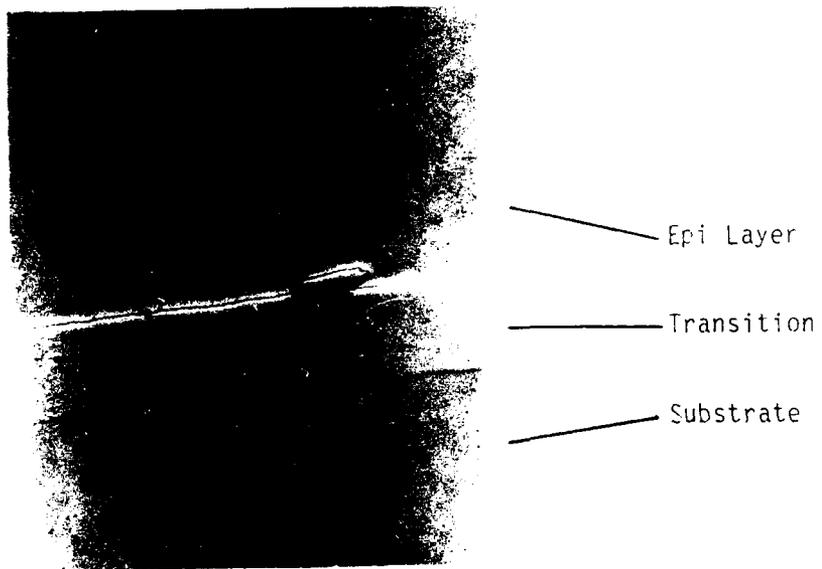
Surface Topography

(Magnification 100x)

Figure 7

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at 200X using DIC. This featureless surface was typical of all surfaces until the thicker samples were evaluated. The surface roughness seen on the thicker samples can be attributed to the mismatch in the cell parameters measured in the epitaxial film and substrate. This information will be discussed in the next section.

With the techniques used for this section of evaluation, the ICB method produces uniform film thickness and very smooth surfaces. Also this method has a unique property of cleaning the interfaces prior to deposition in order to produce a surface that is atomically clean.

b. Structural Properties

The structural properties of the samples were measured in two ways: x-ray diffraction and SEM channeling. The reason for the extensive structural analysis was to determine if the films were single crystal. Some of the electrical data measured were abnormal, thereby suggesting the possibility of amorphous or polycrystalline films. The first study was done with a scanning electron microscope (SEM). Using the SEM, electron channeling patterns are generated as a result of the crystallographically dependent electron backscattering from the surface regions of crystalline material. The scattering of a 30 KeV beam is primarily from the 500-1000 Å region. Using this method, the

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crystalline properties can be evaluated across a given sample by measuring the channeling patterns generated and comparing them. The whole of each surface examined gave the same (100) patterns as shown in figure 8. At even lower energies, 5 KeV, (100) patterns were also obtained for the surface images.

To further examine the problem, a sample was cleaved, and the (110) planes were evaluated. The patterns obtained were (110) at the surface, proving the single crystal nature of the film as shown in figures 9 and 10.

One interesting aspect of this evaluation was the cross section shown in figure 11. The epi layer is much brighter than the substrate. This is due, possibly, to a difference in electrical conductivity. Since the SEM electrons are being introduced into the lattice by ionization, the illumination could be from re-radiation of the secondary electrons from native defects in the films. The x-ray diffraction technique was used as a cross check to measure the single crystal nature of the film. Also, the x-ray diffraction work can be used to determine unit cell parameters using the low incident angle technique. In the two x-ray traces seen in figures 12 and 13, the two effects can be seen. On sample #20, the presence of the second peak on the 422 reflection is evidence of different cell sizes with the

29

Figure 8

Typical Electron Channeling Pattern
Electron Beam Energy   35 KeV

The clarity of the pattern and the presence of high order lines
indicates the perfection of the crystal surface region. The pattern
can be indexed as a (100) orientation.

30
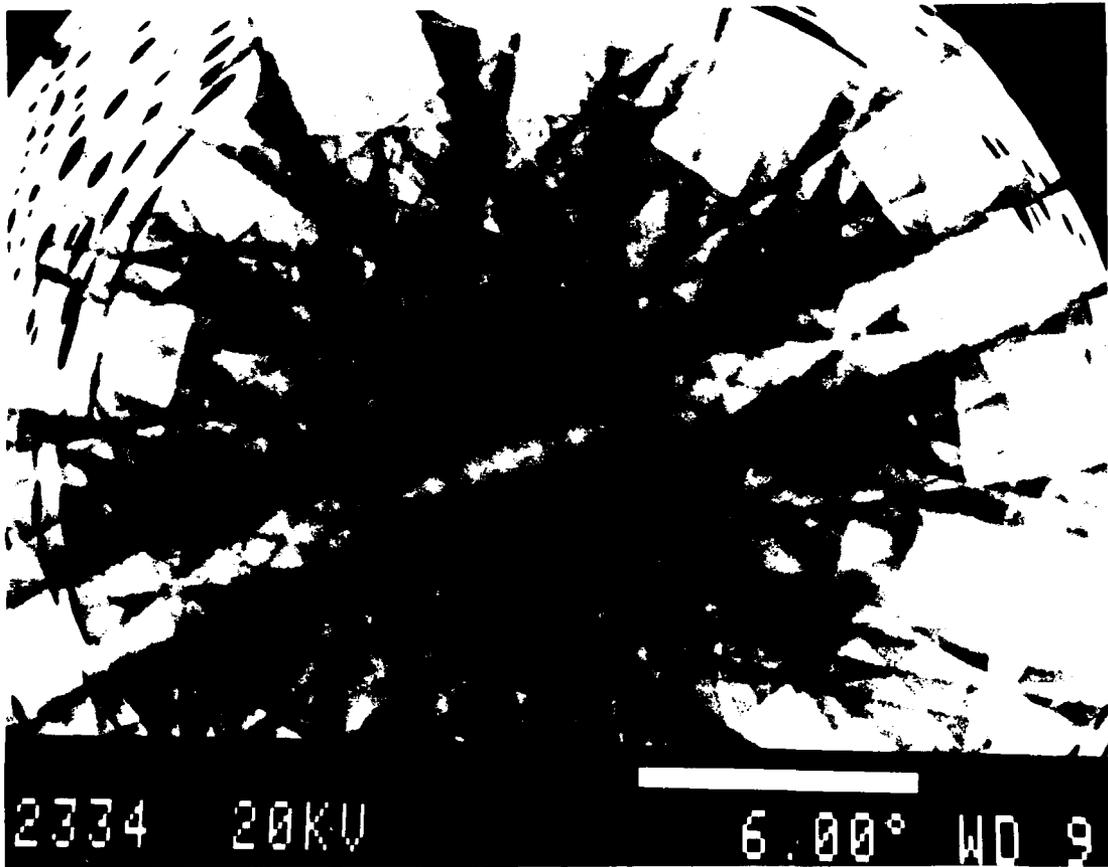
Figure 9

(110) Zone Axis Channeling Pattern from Cleaved
Cross-Section of Sample #015.

31
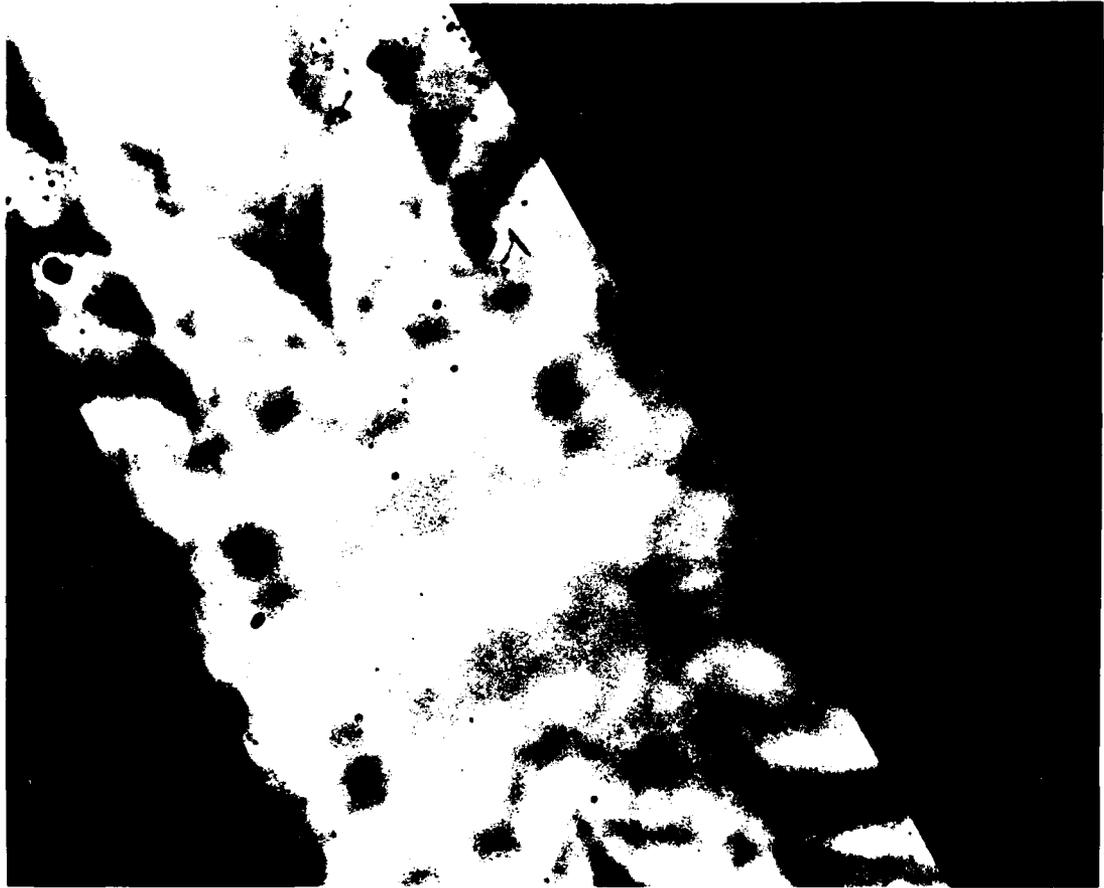
Figure 10

Hybrid image of the cross-section of sample #015. The superimposed
channeling pattern corresponds to the center part of the (110) pattern
shown in Figure 2.

32

Epitaxial
Layer

Substrate

Figure 11

SEM image of cleaved cross-section of sample #015 showing the epitaxial
layer. Note the high secondary electron emission.

33

Figure 12

X-ray Diffraction Trace
Sample Number 20

Diffraction Conditions:
    422 Reflection @ 5 degrees
    (Glancing Angle)
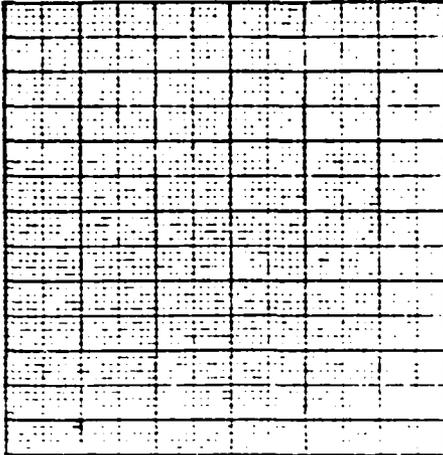
Figure 13

X-ray Diffraction Trace
Sample Number 33

Diffraction Conditions:
    422 Reflection @ 5 degrees
    (Glancing Angle)
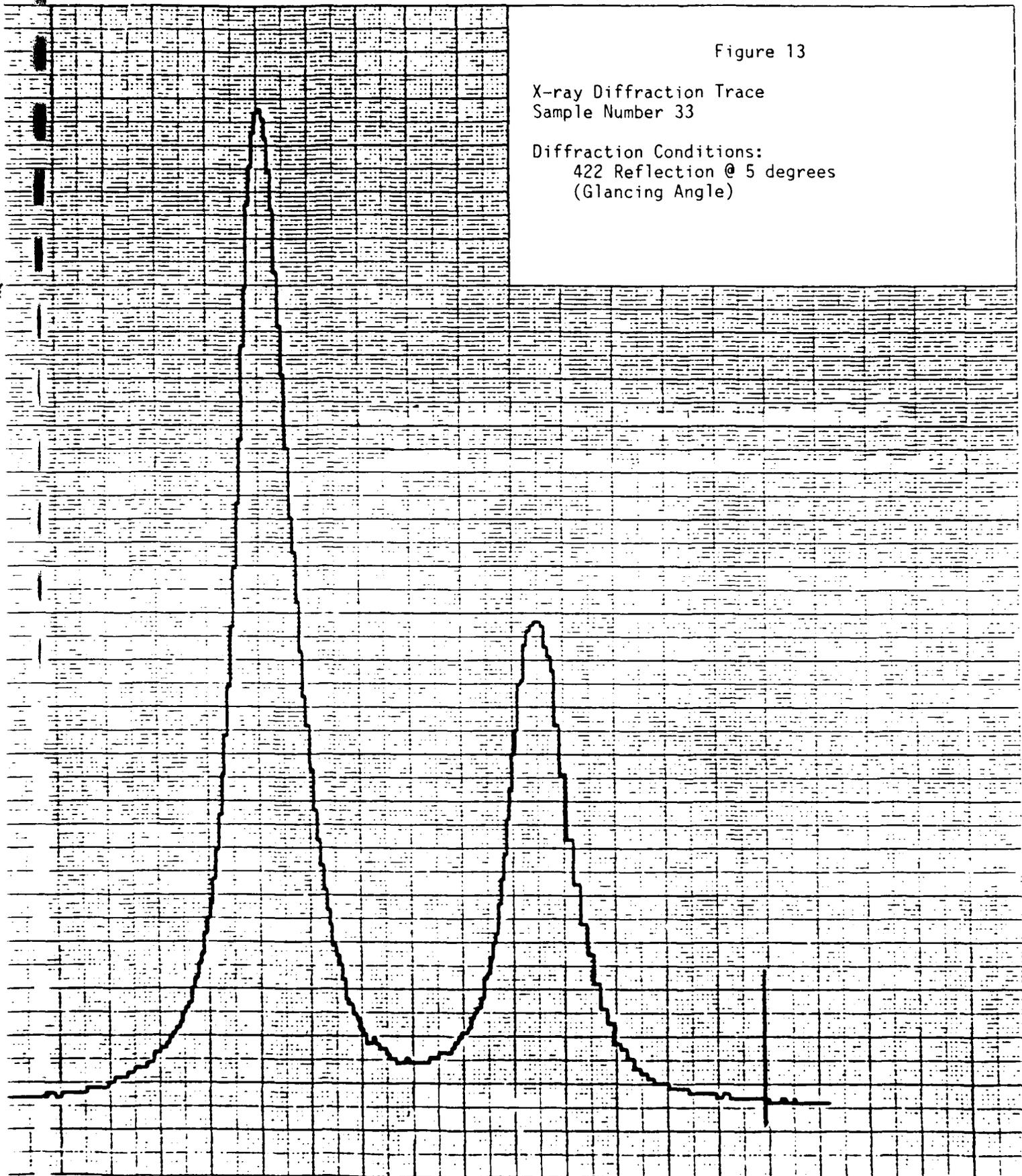
surface having a cell value of 0.1% less than the substrate. By contrast, sample #33 shows a well resolved 422 reflection with 109 seconds as the width at ½ max and an intensity ratio of 218/14 = 15 in arbitrary units. This ratio combined with the narrow reflection indicates a well defined single crystal with lattice parameters matching the substrate. Sample #33 had arsenic pressures of 13 x 10^{-4} Pascal as opposed to 8 x 10^{-4} Pascal for #20. Apparently the presence of the extra arsenic allowed the formation of the correct cell size. Other scans of the 511 and 422 peak show the films to be single crystal.

c. Auger Analysis

Characterization of the samples was done on the (100) surface as well as the (110) faces of the wafer using a Perkin - Elmer Phi-600 Scanning Auger Microprobe. For the surface analysis, the samples were raster scanned in a number of places on the wafer with each analysis done after sputtering 80-100 Å of material away. A 3 KeV accelerating voltage with a 10na current was rastered over a 100μm x 100μm area as well as a 10 KeV accelerating voltage with 10 na. An Ar^+ ion beam was used to etch the (100) surface at a rate of 14 Å/second. The change in surface spectra versus depth can be seen in figures 14, 15, 16, and 17.
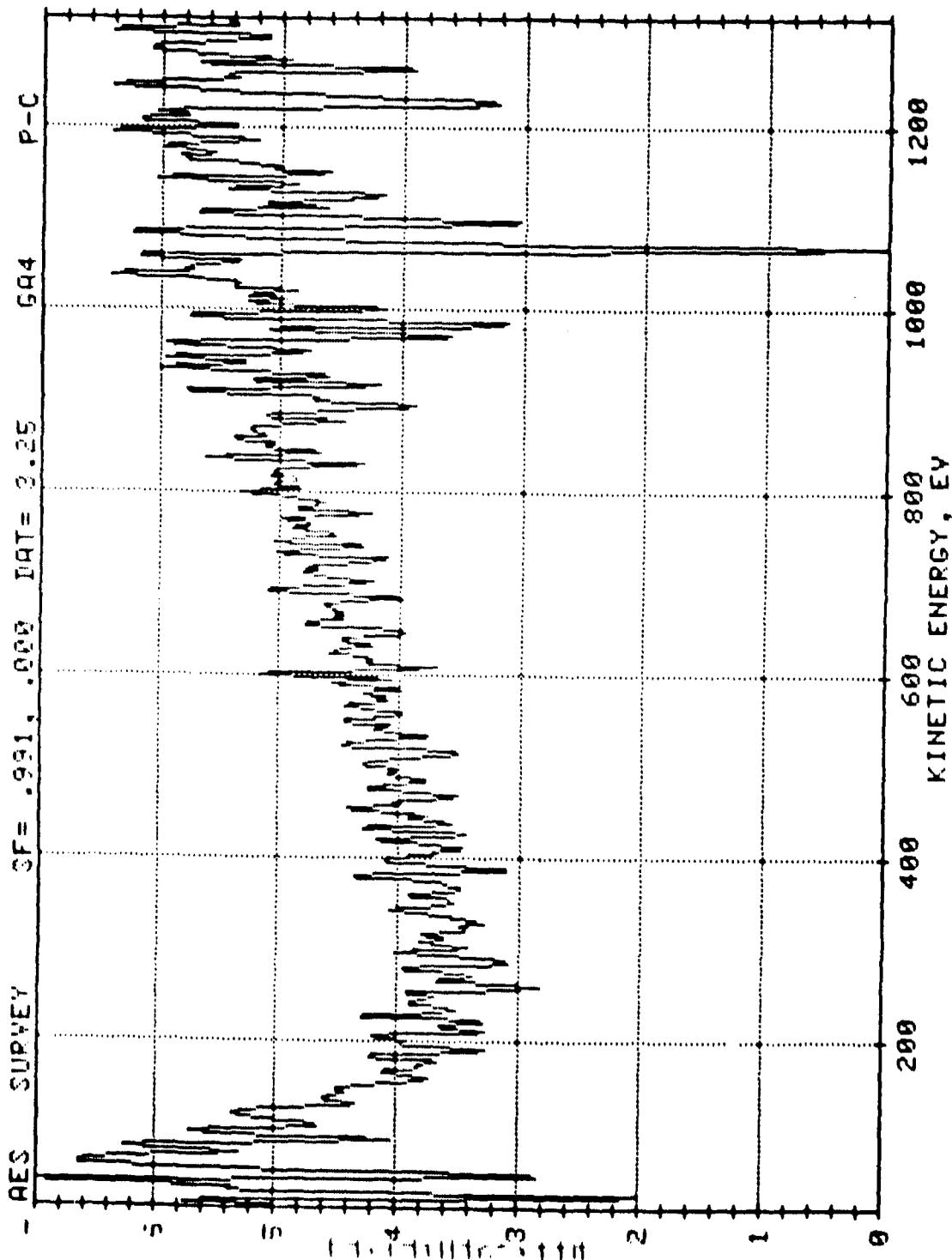
Figure 14

Auger Spectra of a GaAs Substrate on the (100) Surface Prior to Etching
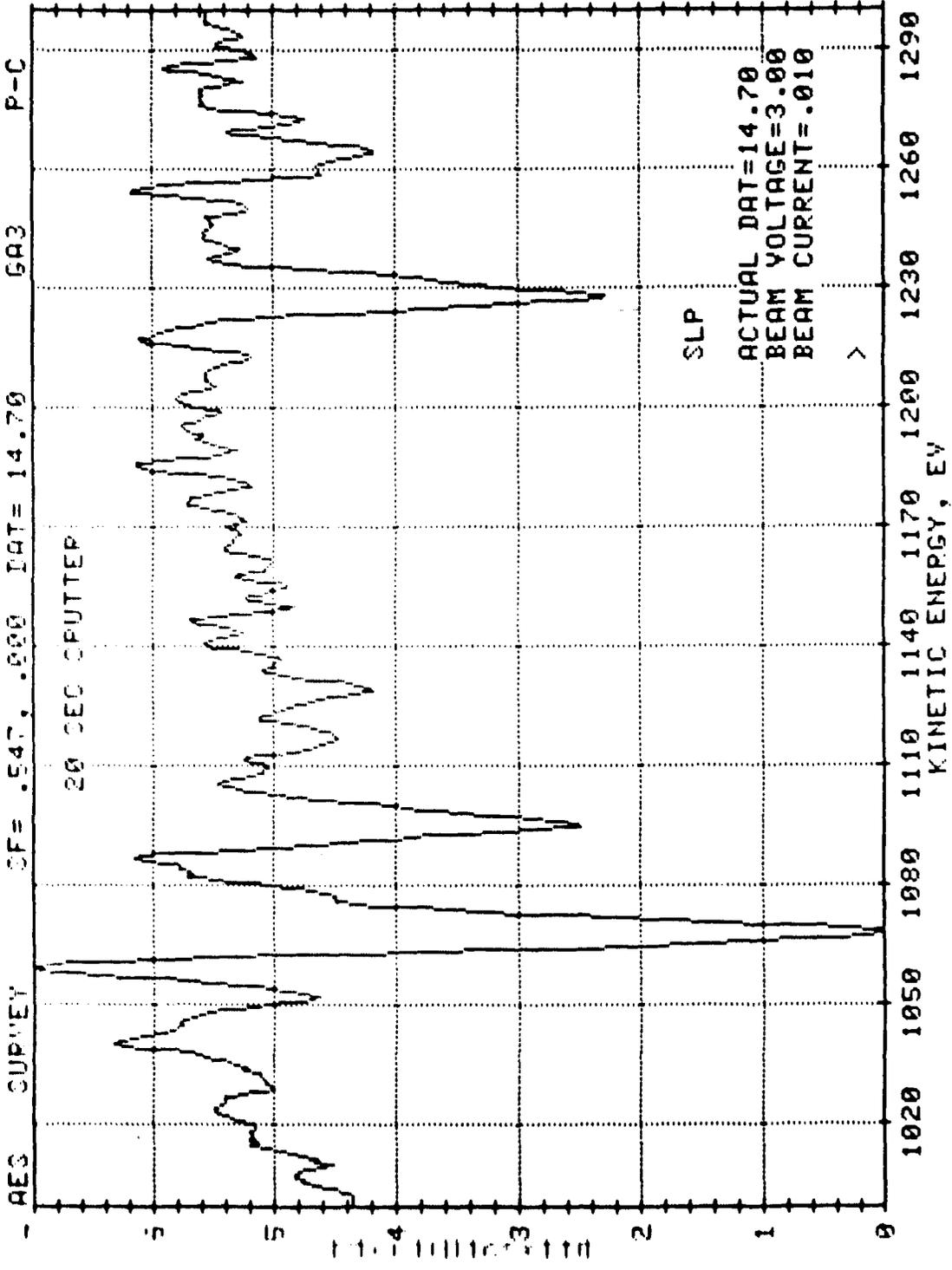
Figure 15

Auger Spectra of a GaAs Substrate on the (100) Surface after 20 second Sputter Etch
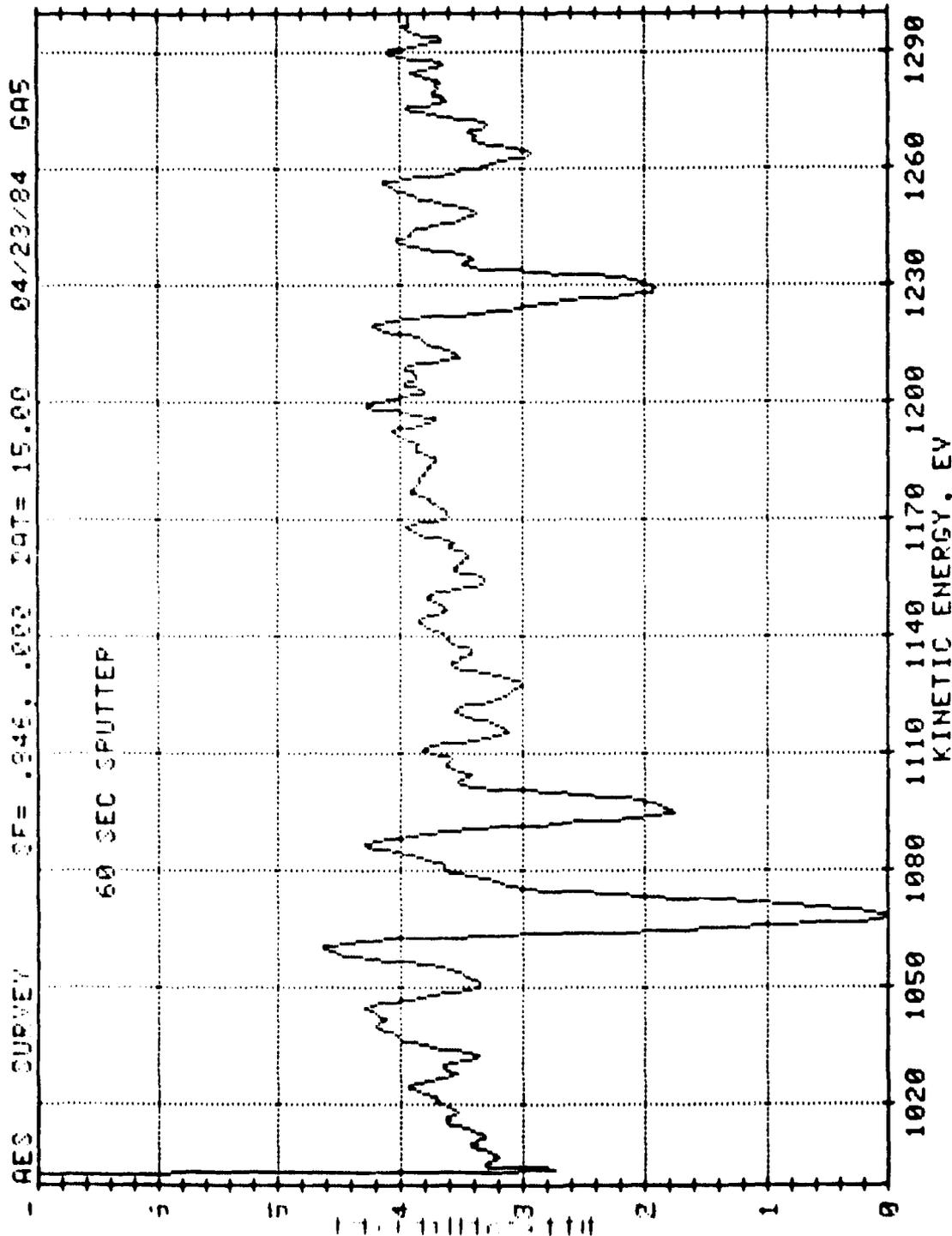
Figure 16

Auger Spectra of a GaAs Substrate on the (100) Surface after 60 second Sputter Etch

39
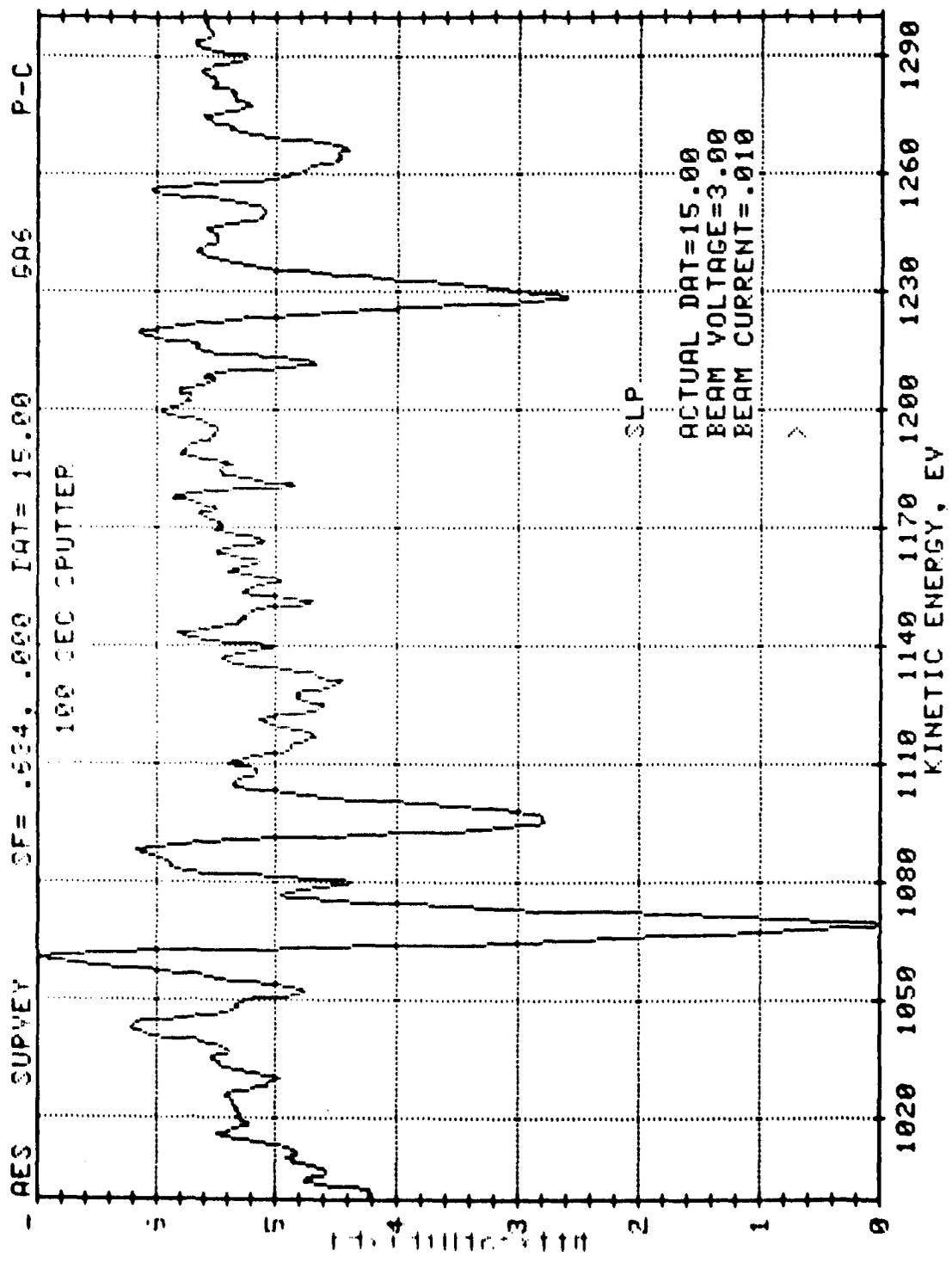
Figure 17

Auger Spectra of a GaAs on the (100) Surface after 100 second Sputter Etch

40

A second set of experiments was also run to characterize the (110) face. In this work a sample was fractured in vacuo, and the freshly exposed surface was evaluated. By this method, the surface film was clearly visible, and the substrate and epi film could be analyzed independently on the freshly exposed surface. For this analysis, a 60 Å spot size was used. The intensity ratio of the $GaL_3M_4M_4$ peak (1228 eV) to the $AsL_3M_4M_4$ peak (1329 eV) was found to be 1.38 for the epi film compared to the substrate. This compares to the value of 1.41 reported by Van Oostrom[6]. To determine elemental concentrations from the peak intensities, relative elemental sensitivity factors must be defined based on a sample matrix of empirical value. Van Oostrom has determined these factors (5 KeV incident beam energy). For the samples evaluated in this work, the following data were obtained for the (110) faces. Each point was corrected using the factors determined by Van Oostrom: The Auger scans for these values can been seen in figures 18 and 19.

Substrate	49.5±0.5% Ga	50.5±0.5% As
Epi	48.3%	51.7%

Although the (110) face is expected to contain equal concentrations of Ga and As atoms, a variation of ±10% is reported due to differences in step densities[7]. Therefore, the results agree very well for both epi and

41
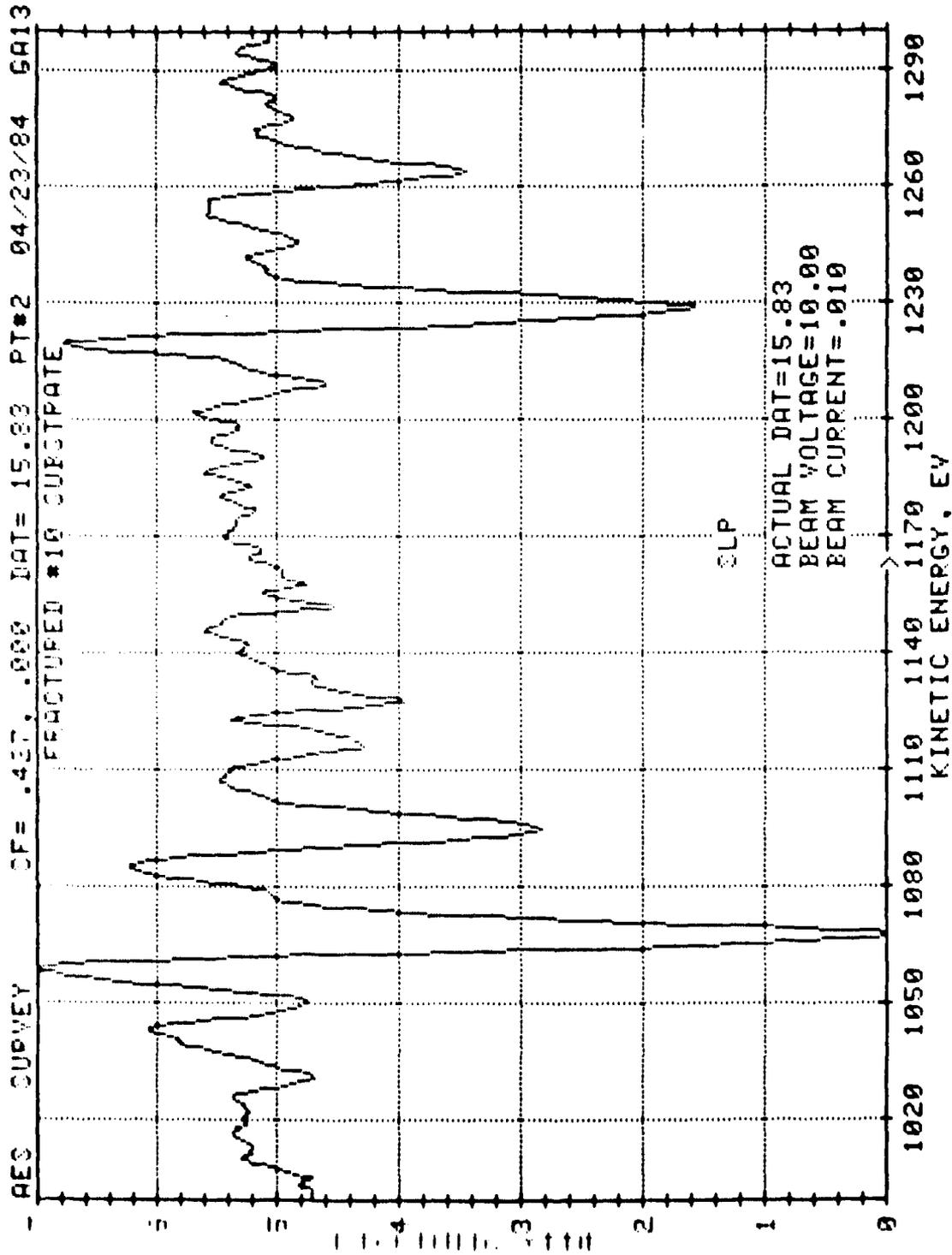
Figure 18

Auger Spectra of Sample No. 10 Substrate on the (110) surface cleaved in vacuum

42
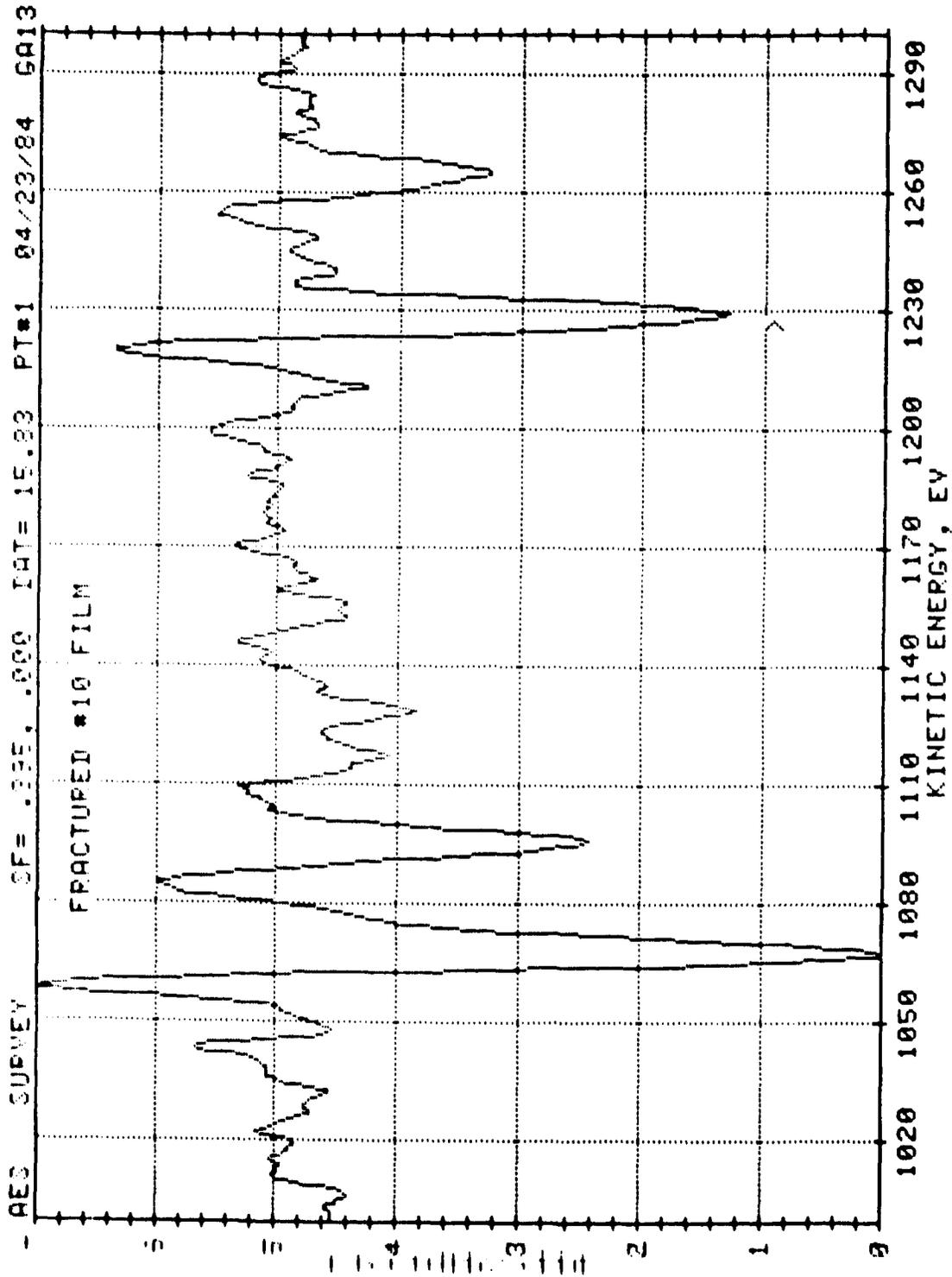
Figure 19

Auger Spectra of Sample No. 10 Epi Film on the (110) Surface cleaved in vacuum

substrate.

Previous work on (100) faces has shown a preferential etching of the arsenic yielding an intensity ratio of 1.8 for Ga/As and a stoichiometry of Ga=56% and As=44%[8]. The results obtained on samples grown in this work were:

	I_{GaLMM}/I_{AsLMM}	Ga(At%)	As(At%)
Sample #10	1.90	57.4	42.6
#13	2.02±0.19	58.8	41.2
#15	1.81	56.3	43.7
Substrate	1.95±0.07	58.1	41.9

The Auger spectra for the samples can be seen in figures 20, 21, and 22. In-situ Auger analysis would better resolve surface stoichiometry variations since, once the surface is exposed to the atmosphere, hydrocarbon contamination and surface oxidation alter the concentrations. However, this option is not available on the existing hardware. Thus, the in-vacuum cleaving of the sample used in these studies should produce the most accurate alternative, and this work showed the stoichiometry of the substrate to equal the epitaxial film.

d.   Electrical Evaluation : Hall Data, Capacitance/Voltage (C/V) Profiles, and Photoluminescence (PL) Scans

The C/V data for the samples was not meaningful. In each case, there was no change in capacitance as a

44
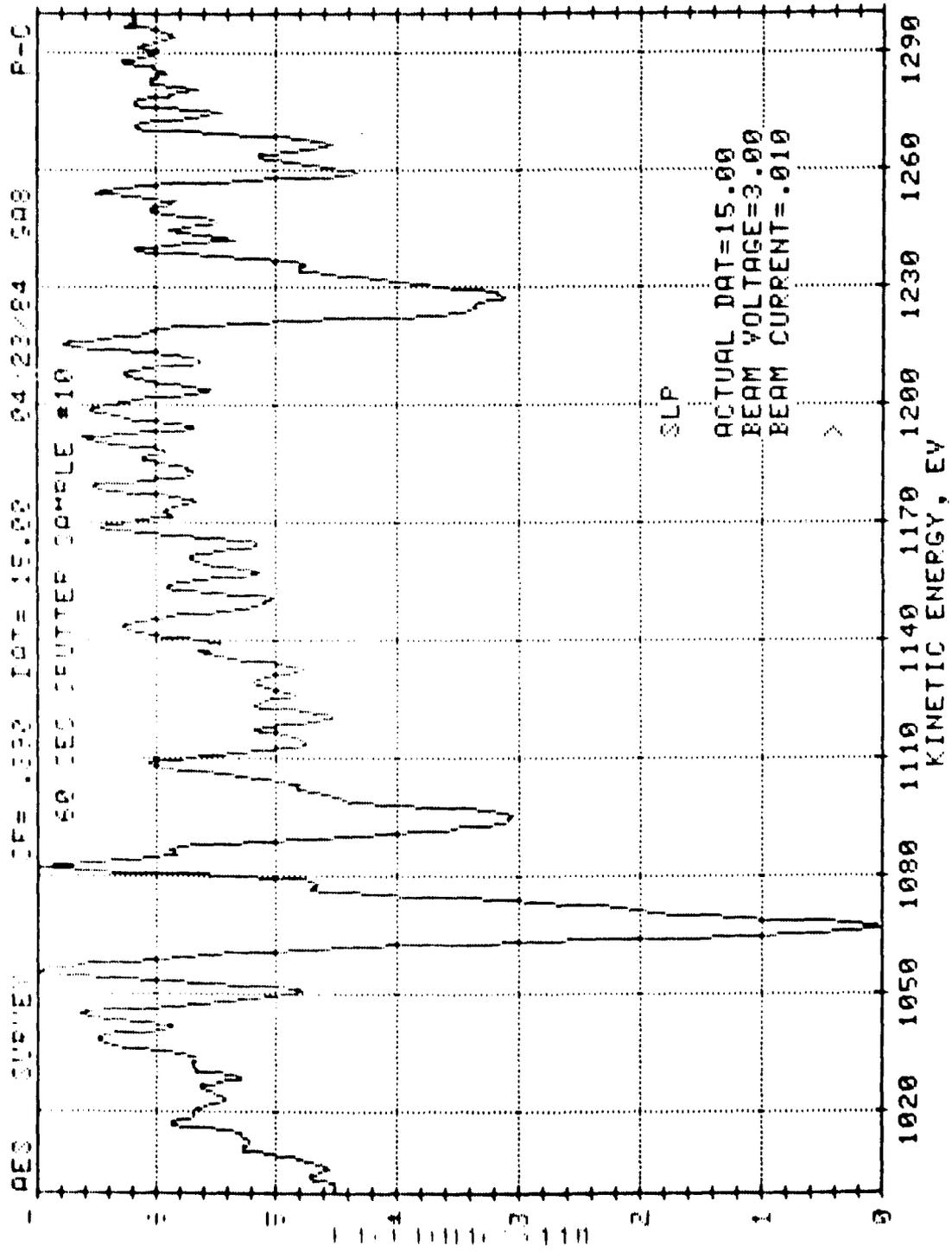
Figure 20

Auger Spectra of Sample No. 10 Epi Film on the (110) Surface cleaved in vacuum

45
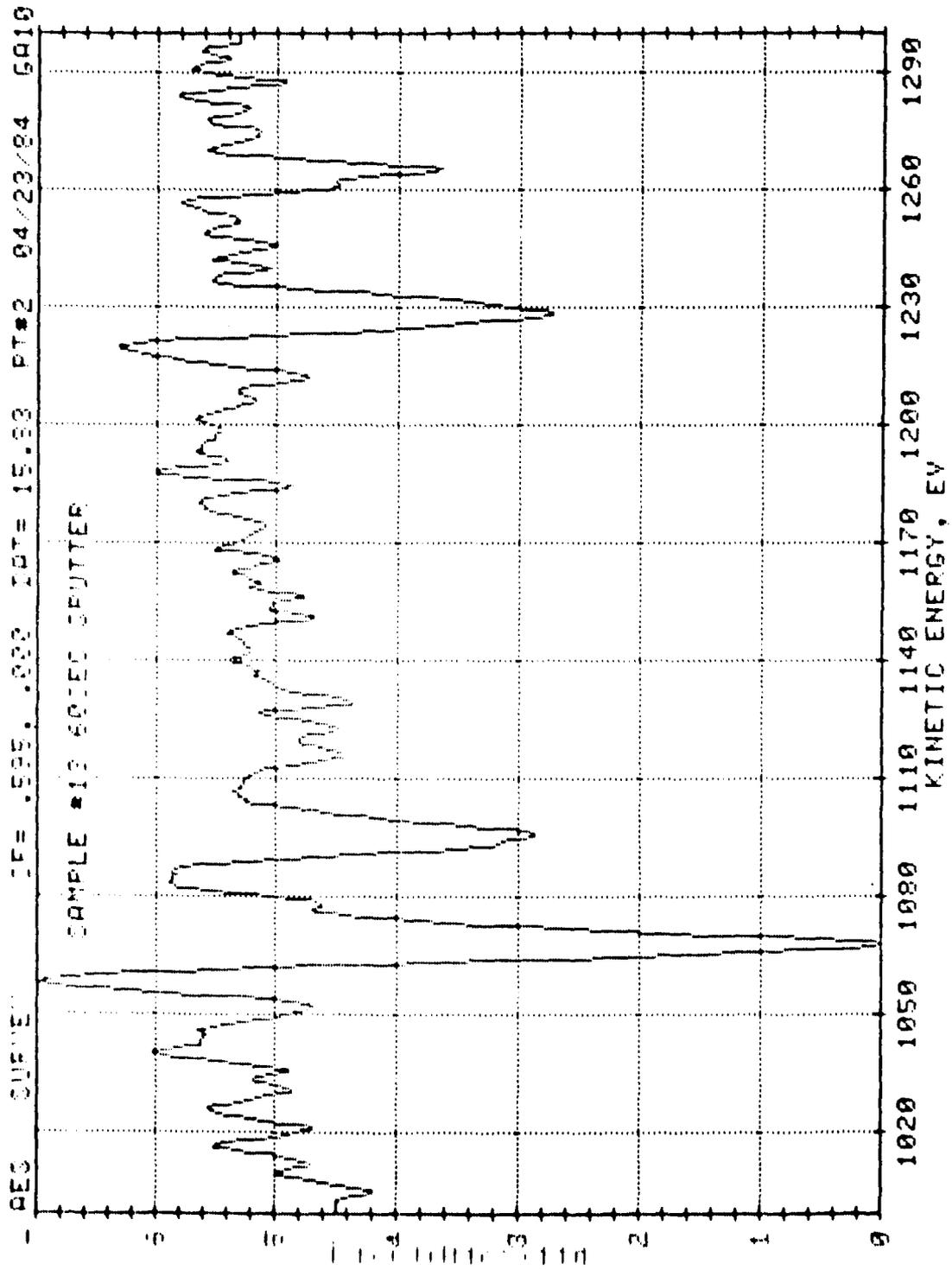
Figure 21

Auger spectra of Sample No. 13 Epi Film on the (110) Surface cleaved in vacuum
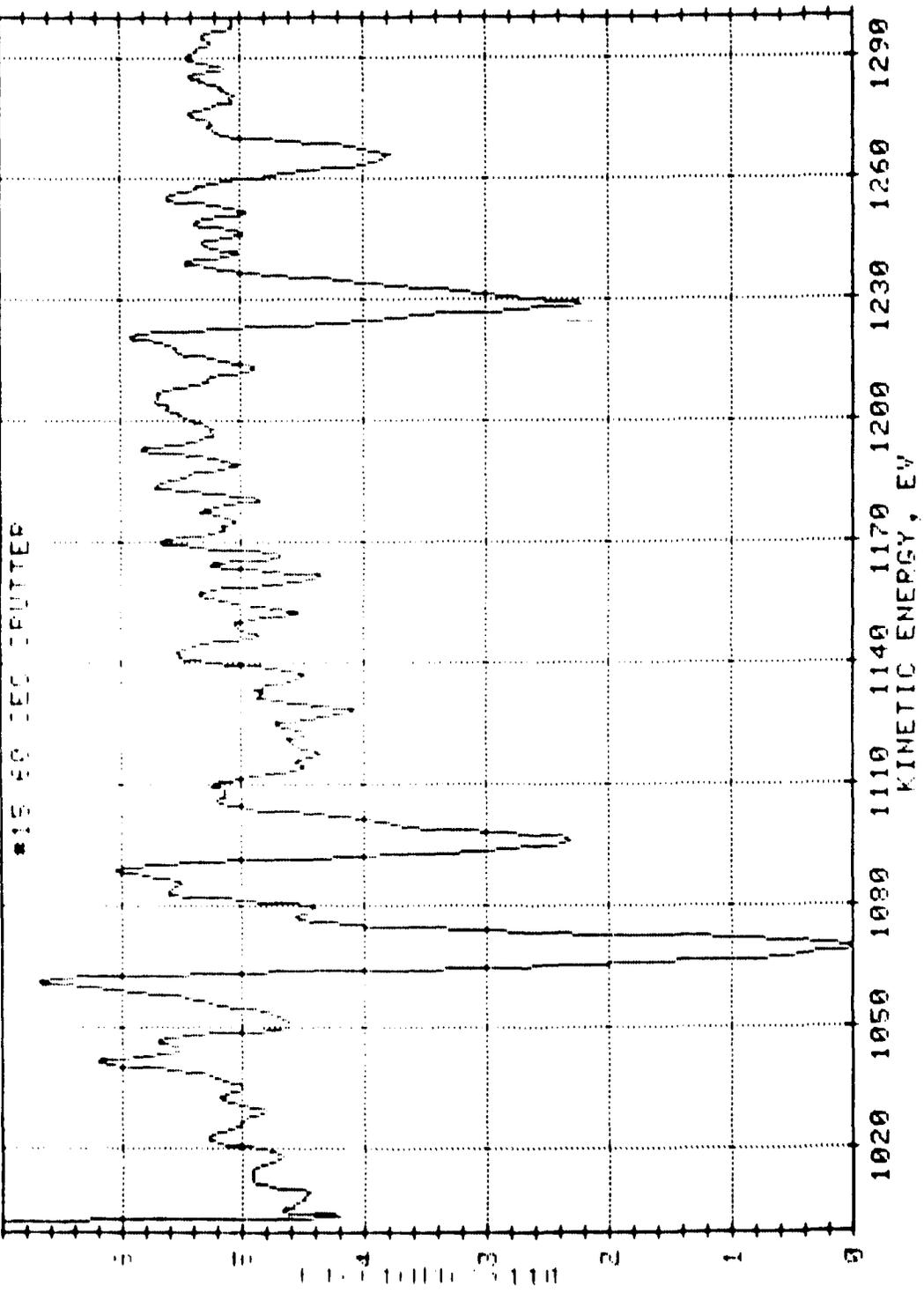
Figure 22

Auger Spectra of Sample No. 15 Epi Film on the (110) surface cleaved in vacuum

function of voltage. This was due to the high resistivity of the epi film which caused the depletion layer to extend beyond the epi film and into the substrate. A mercury probe was used for these measurements, and a standard film was used to calibrate the system. When one compares the Hall data with the lack of a meaningful C/V profile, the explanation is very reasonable. Until the films are lower in resistivity and thicker, the C/V technique will be of no value.

The Hall data collected on all the samples shows three very consistent trends:

1. High resistivity of films
2. n-type conductivity
3. very low mobility.

The samples grown in the second series of runs in March were found to have electrical resistivities of 10^3 ohm-cm, independent of thickness. The last three samples with higher arsenic pressure show low mobility, but this demonstration of mobility is accompanied by lower resistivity, 400 ohm-cm, and a more ohmic nature. When the leads are reversed on these samples, the values are the same in both directions indicating a very ohmic contact.

Photoluminescence was evaluated on these samples but was very inconclusive although these results are quite consistent with the C/V and Hall data. Because of the lack of meaningful data, PL was not used extensively. Pl scans for samples #13, 15, 16, and 17 are shown in figure 23. The general conclusion is that the overall electrical quality of the film is quite poor.

4. Conclusion

Phase I results demonstrated that the Eaton ICB machine can grow films of GaAs that are single crystal and at temperatures below 400 °C. The technique is repeatable from run to run and produces films that are of uniform thickness across samples 0.7" x 0.7". However, the low mobilities and high resistivity indicate major problems. Subsequent work at Eaton, combined with the data gathered in this work, possibly offer an explanation for these results.

Work done several years ago by Schiller et. al.[9] studied the predominant ion actions within different energy regions. A graphic showing these conclusions is presented in figure 24. The interesting section is that from 1-1000 eV per ion. In this region, material with activated centers, i.e. - charge defects, structural defects, and material defects is produced. If there were no clusters found or only clusters with approximately 10 atoms, the average kinetic energy of the accelerated ions (assuming 3 KeV acceleration potential)

49
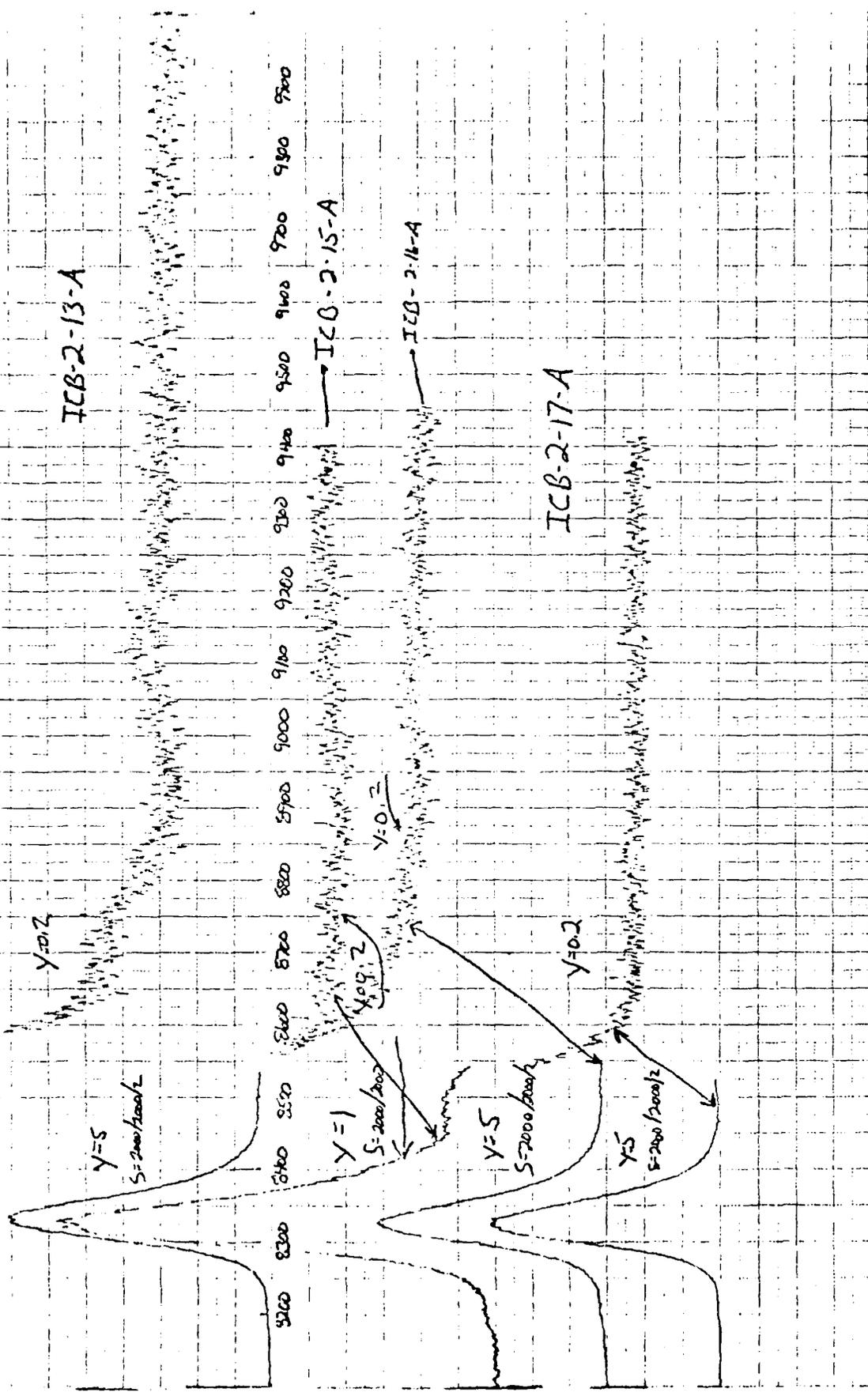
Figure 23

Photoluminescence Spectra of Samples 13, 15, 16, and 17

Figure 24

Predominant Ion Actions Within Different Energy Range

Thermal activation of condensation - Increasing of migration

Desorption of impurity atoms

Creation of activated centers (charge defects, structural defects, material defects)

Sputtering of impurity atoms, resputtering

Implantation

ION ENERGY (ev)

10^{-2} 10^{-1} 1 10 10^2 10^3 10^4 10^{-5}

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would be in the range of 300 eV. In this range, one would
expect single crystal material, but with poor electrical
properties due to the multitude of native defects created by
the presence of such high energy ions. This structure would
indeed be expected to have very low mobility and high
resistivity. Even on those samples having a correct lattice
constant, the impinging energy is great enough that the
defect is still the dominant mechanism.

Work at Eaton subsequent to the last set of experiments has
shown that there is little or no cluster formation from
crucibles that are not uniformly heated. Rather, droplets
accumulate in the crucible, particularly at or near the
nozzle area. This is consistent with the observations
previously made when the gallium source cap was removed.
Further work at Eaton with the newly designed radiant heated
sources, showed enhanced heating uniformity and, indeed, the
formation of clusters. However, the cluster size was still
less than the 1000-2000 atoms Takagi quotes for standard
cluster size.

As with any new hardware and technology, a great deal was
learned from a few experiments. The limited results obtained
appeared very encouraging as to the viability of ICB for
producing acceptable quality single crystal epi films.
Subsequent work on the machine coupled with improved process
understanding should move the technology rapidly.

III.   Phase II Summary

A.   Machine History

The history of the current hardware can be traced to the first generation machine that was used to produce the data generated under the Phase I Contract No. F33615-83-C113 which was awarded on September 16, 1983.

While the data from Phase I showed some promise that the process was viable, there were numerous hardware problems that had to be solved prior to further progress on development of a process.

Some of the problems included:

- Excessive heating of the sources and surroundings due to the close proximity of the substrate heater.

- Common acceleration of all sources which prevented true independent control over each material being evaporated.

- Excessive gallium arsenide deposits on and around the sources, most likely resulting from inappropriate nozzle geometries.

- Extremely poor source temperature control due to the lack of a feedback system and virtually no control on the low temperature arsenic source resulting from the use of e-beam bombardment type heaters.

- Very unstable ionization and acceleration power supplies.

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All of these issues, plus several other concerns, were addressed in the design and construction of a second generation machine, which is where the current Phase II Contract begins.

The machine that was to be used for the Phase II research was built around the same basic vacuum system as that used for Phase I, but with several modifications to the source and substrate areas. These include radiant type heaters on each source, three totally independent sources using more stable power supplies, thermocouple feedback loops on each source for accurate temperature control, and relocation of the substrate area to a distance of approximately 24 inches from the sources. This machine was delivered to Epitronics in May, 1985. It was installed and made ready for start-up the following month. In July, a factory representative was sent to Epitronics for start-up.

During start-up, many small problems were identified and fixed, but on the third day of start-up the vacuum system controller failed and had to be replaced. At this point the start-up could not be continued.

Also, during this time frame, the first evaporations were done. Upon opening the chamber the first signs of improper nozzle/source design were apparent. The pictures on the following page, in figure 25, show the contamination that occurred on the arsenic accelerating electrode after one evaporation. The gallium source

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Arsenic Source
with Original Nozzle

Figure 25

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demonstrated similar contamination.

Once the new vacuum controller was installed, Epi-Tech personnel completed the remainder of the machine start-up.

The next order of business was to test the vacuum system integrity and establish a system baseline, but upon repeated cycling of the vacuum system, several leaks to atmosphere, as well as water leaks from the internal cooling lines, were discovered.

The atmospheric leaks were relatively easy to locate, using a Helium Leak Detector in combination with the ICB System RGA unit, and in most cases were simply dirty or slightly scratched o-ring sealing surfaces.

With the atmospheric leaks fixed, the process of tracking down the water leaks began. These were eventually isolated to the areas where a stainless steel fitting was joined to a copper cooling coil. These joints were brazed instead of welded, and when the unit was thermally cycled the brazing developed hairline cracks due to the difference in expansion coefficients between the copper and stainless steel, thus producing leaks.

Also, during this period of time, it became very apparent that a hot water recirculating system was necessary to help reduce the quite lengthy bake-out times currently required to reach a reasonable base pressure.

With the leaks fixed and the hot water system installed, some

initial electrical check-outs revealed missing parts on both the deposition rate monitor and Faraday cup; and in addition, the entire system demonstrated large calibration errors.

While these problems were being fixed by local factory personnel, the process of calibrating the actual substrate temperature to the reference flag was started.

It was during this process that two significant problems were discovered. The first problem dealt with the fact that the substrate temperature would just barely reach 300 °C at which point severe heating of the top chamber occurred. This was fixed by relocating the substrate heater assembly to a position closer to the substrate. This partially reduced the top chamber heating but the substrate temperature would still only reach 375 °C with any stability. At this point it was decided to try this arrangement since to correct the problem would have required a major design change and a serious delay of the research.

The second problem was the appearance of a water leak on the RGA which continued to increase in magnitude the longer the substrate heater was in operation. This problem was difficult to isolate since the top chamber contains many welded cooling jacket seams that could expand as they get hot, in addition to four swagelock type fittings on the cooling lines to the deposition rate monitor sensor. These likely candidates were eventually eliminated by helium leak checking in the case of the cooling jacket seams and switching to a fully welded bakeable type unit in the case of the

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deposition rate monitor sensor.

With these possibilities eliminated and the problem still persisting, the top to the chamber was removed, at which time it was discovered that the thermocouple and electrical feedthroughs, which were made of a phenolic material, were severely decomposing due to the excessive heating that occurs in the top chamber area when the substrate heater is on. This was fixed by installing fully bakeable type feedthroughs.

Having fixed these problems, the task of calibrating the substrate temperature, as well as calibrating the evaporation rate versus source temperature, in each source, continued.

As the process calibrations and testing proceeded, the source hardware used for ionization and acceleration was reinstalled.

The next step was to try just ionizing the material beam, which went relatively well although the sources were somewhat unstable.

Finally, the accelerating potential was applied, at which point it was discovered that the system would only support one kilovolt of potential out of a possible ten kilovolts before arcing and shutting down the system. This problem was finally traced to improper high voltage cables from the power supplies to the sources, i.e. - the cable insulator would not support the high potential, thus allowing the conductor to arc to the cable shield.

At this point, with each source able to fully operate

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independently, several source design problems were starting to become apparent.

The first of these problems is the severe source coating that occurs during an evaporation which, as previously stated, is most likely due to improper nozzle geometry. This coating is also indicative of another problem, namely arcing within the source at low accelerating potentials. This is most likely due to a locally high vapor pressure in the vicinity of the ionizer and accelerator electrodes. This condition prevented the source from being operated at accelerating potentials above about 2.5 kV and also caused the ionizer filaments to burn-out after very short periods of operation.

Another problem was the isolation of each source, i.e. - attempts to use two or more sources simultaneously revealed severe cross talk between the sources which prevented independent operation. The reason for this appears to be the lack of a reference electrode above the source. This is allowing the field lines to spread out over the entire chamber and couple to the neighboring sources.

Also, it would seem that the basic design of the ionizer is not appropriate for providing a reasonable degree of ion production in the beam. This is supported by the fact that during operation a maximum of 40-50 mA of current is all that can be extracted from the ionizers which produces so few ions that there is no reading on the Faraday cup display.

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Finally, it appears that the mounting of each source is at such an angle as to cause the beam centerline to be removed by approximately six inches from the center of the substrate. This is possibly the reason for the lack of a reading on the Faraday cup display and is most likely the reason for the reduced growth rates in runs where a columnating (deLeval type) nozzle was used.

Having identified the aforementioned problems and considering their magnitude, it was decided that the best possible approach was a complete redesign of the source ionization and acceleration sections. In addition, a complete redesign of the substrate heater area was decided upon to address the problems of low substrate temperatures and excessive heating of the top chamber. At present, both of these redesigns are nearing completion.

B. Process History

The first samples of gallium were grown on August 27, 1986. These samples were grown on glass slides and were intended as calibration samples for gallium evaporation rates versus source temperature.

During this period of time the arsenic source was started and run independently to determine arsenic evaporation rates versus source temperature.

Calibration runs continued until the first gallium arsenide was grown on a glass substrate on September 16, 1986.

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From this point a few more gallium arsenide calibration runs were performed with the sources in an "MBE like" configuration, i.e. - large crucible opening and no ionization or acceleration hardware installed.

Finally, on September 18, 1986, the first growth on a Gallium Arsenide substrate was performed with the substrate at ambient temperature. Following this run was the first run with an elevated substrate temperature on September 24, 1986, which is listed as Run No. 002.

The runs following No. 002 up through Run No. 008 were all run in an "MBE like" configuration and attempted to establish a baseline of information at various V/III ratios and substrate temperatures.

The next series of samples, Run Nos. 009 through 030 were generated using deLeval type nozzles. The objective of this series of runs was to produce a film of specular quality by varying items such as, nozzle size, substrate temperature, ionization and acceleration levels within the hardware limitations, and V/III ratio.

On May 27, 1987, Sample No. 030 was grown after which the failure to produce a specular sample, accumulated data, and general observations indicated that a major hardware redesign was necessary. As stated in the previous section this is currently under way.

C. Description of Machine Operation

The basic process began with a thorough bake out of the vacuum chamber each time the chamber was opened for source replenishment, which until the last few runs was after each run.

The first step in this bake out was to pump the system down to an operational vacuum level, approximately 1×10^{-4} torr. Next, the hot water recirculating system was turned on. This supplied hot water to the chamber wall jacket and the cooling lines on the sources. Once the chamber had reached an elevated temperature approximately 130 °F, and with the vacuum level at or below 1×10^{-4} torr, the substrate heater and source crucible heaters were turned on at low levels.

These levels were such that the evaporant material in the source crucible was maintained at a temperature just below that required to generate any substantial vapor pressure.

The system was allowed to operate in this mode until the vacuum level had dropped to below 1×10^{-6} torr. At this point, the source crucible heaters, substrate heater, and hot water recirculating system were turned off and the machine allowed to cool for a period of two hours. After cooling down, the chilled water recirculating system was turned on and the source crucible heaters and substrate heater were turned back on at the same low levels previously mentioned. The machine was allowed to stand overnight in this mode until approximately one hour prior to

starting a run. At this point the liquid nitrogen baffle over the diffusion pump was turned on.

This overall procedure takes approximately twenty-four hours from the time the chamber door is closed, and produces a system base pressure between 5-7 x 10^{-8} torr.

With the system now at base pressure it is ready to accept a sample for growth.

In preparing the samples, the first step was to clean the substrate in a ten percent by volume HCl/ethanol solution.

This was followed with a D.I. water rinse, ethanol rinse, and a final blow dry with dry filtered nitrogen.

The substrate was next placed on a stainless steel substrate carrier and a 1.5mm thick molybdenum backing plate was attached using a thin liquid gallium film as the heat conducting medium.

This mounting procedure is essentially the same as that used in Phase I and pictured in figure 5D. The only difference is that a molybdenum backing plate was used in place of stainless steel.

Finally, the substrate carrier with the substrate was placed into an ambient temperature load lock. This load lock was pumped down to less than 7 x 10^{-7} torr at which point the substrate was transported into the main chamber for growth.

Once the substrate was in position, the shutter on the arsenic
source, already at a stable operation temperature was opened
exposing the substrate to a flux of arsenic while the substrate
was being heated up to growth temperature. This procedure was
used in order to provide some degree of in-situ cleaning while
preventing the exposed surface from becoming arsenic deficient
during heat up.

Upon stabilization of the substrate temperature, the shutter on
the gallium source, already at a stable operating temperature was
opened and growth of the gallium arsenide film started.

When the growth was complete, the shutter on the gallium source
was closed, the substrate heater turned off and the sample was
allowed to cool down under a flux of arsenic until the temperature
was below 200 °C, at which point the arsenic shutter was closed
and the sample transported into the load lock for the remainder
of the cool down.

The sample was subsequently transported out of the load lock,
dismounted from the molybdenum disk, the excess gallium wiped off
and the remaining thin gallium film removed by soaking the sample
in a warm twenty percent by volume solution of hydrochloric acid
in D.I. water.

D. Run Parameter Summary

The run conditions presented in Table III on the following pages
can be split up into two general categories. The first category

deals with samples numbered 001 thru 008. These samples were ones that used a large orifice crucible with no ionization or acceleration hardware installed. This was considered to be an "MBE like" mode of operation and was used to generate a baseline of data.

The second category concerns the remaining samples numbered 010 thru 030. These samples were produced using various nozzle configurations, described in appendix A, in combination with some ionization and acceleration.

As can be seen from the table, there were only a few runs which had ionization or acceleration applied to either the gallium or arsenic source. This was primarily due to the mechanical and electrical problems encountered with the sources as previously discussed.

Also, the thickness of the majority of samples was difficult or impossible to measure. This could have been due to a close match between the substrate character and film character or the lack of growth. The first appears to be the most likely since the surface pictures and TEM pictures indicate that a film was deposited.

TABLE III

Run/ Sample No.	Nozzle No Ga	Nozzle No As	Substrate Temp. °C	As/Ga Ratio**	Ga Ioniza-tion mA	Ga Accel. KV	As Ioniza-tion mA	As Accel. KV	Growth Time min	Growth Rate* Å/min.
001			A B O R T E D							
002	01	01	375	5:1	-	-	-	-	480	50
003	01	01	375	7:1	-	-	-	-	420	NA
004	01	01	300	7:1	-	-	-	-	405	22
005	01	01	200	6:1	-	-	-	-	425	14
006	01	01	200	9:1	-	-	-	-	415	19
007	01	01	200	2:1	-	-	-	-	430	NA
008	01	01	100	5:1	-	-	-	-	340	29
009			A B O R T E D							
010	02	02	375	9:1	-	-	-	-	330	NA
011	01	02	300	13:1	-	-	-	-	280	NA
012	03	03	375	3:1	5	-	15	-	390	NA
013	03	03	375	11:1	5	-	15	-	310	NA
014	03	03	375	10:1	-	-	-	-	340	NA
015	02	03	375	47:1	-	-	-	-	285	NA
016	02	02	375	35:1	-	-	-	-	300	NA
017	03	03	375	42:1	-	-	-	-	290	NA
018	03	03	375	50:1	-	-	-	-	320	NA
019	03A	04	375	36:1	-	-	-	-	180	NA
020	03A	03A	350	31:1	-	-	-	-	275	NA
021	03A	03A	325	31:1	-	-	-	-	240	NA
022	03A	03A	300	46:1	-	-	-	-	210	NA

*Growth rate determined from film thickness as measured on an SEM.
**As/Ga ratio determined by weight difference in crucibles from start to end of run using consistant start up and shut down procedures from run to run.

TABLE III (cont.)

| | Nozzle No | | | | Ga | | As | | Growth | |
Run/ Sample No.	Ga	As	Substrate Temp. °C	As/Ga Ratio**	Ioniza- tion mA	Accel. KV	Ioniza- tion mA	Accel. KV	Time min	Rate* Å/min.
023	03A	03A	300	23:1	50	3.0	-	-	300	NA
024	03A	03B	375	37:1	20	3.0	-	-	155	NA
025	03A	01A	375	33:1	30	2.0	-	-	240	NA
026	03A	01A	375	11:1	30	2.0	-	-	150	NA
027	03A	03B	375	N/A	40	3.0	-	-	120	NA
028	03A	03B	375	N/A	40	3.0	-	-	75	NA
029	05B	05B	375	N/A	40	2.5	-	-	70	NA
030	05B	05B	375	N/A	40	2.5	-	-	90	NA

SUMMARY
ICB RUN PARAMETERS

*Growth rate determined from film thickness as measured on an SEM.
**As/Ga ratio determined by weight difference in crucibles from start to end of run using consistant start up and shut down procedures from run to run.

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IV.   THEORETICAL

The following two subsections will present brief overviews of the
principles and concepts behind the design and construction of the
various nozzles used and also the redesign of the sources.

A.   Nozzle Design

The first step in designing an appropriate nozzle for the
evaporation source was to define, generally the type of system
with which we were dealing and the specific reasons for which a
nozzle was being used.

In defining the type of system, the boundaries were restricted to
somewhere just prior to the entrance to the nozzle and just
beyond the exit from the nozzle. In doing this, the nozzle can
now be thought of as having an infinite supply of vapor at some
pressure P and being vented into an infinite volume at some
pressure P_c.

With the above restrictions and assumptions in mind, the nozzle
can now be modeled as though a "real" compressible gas at elevated
temperature were flowing through the nozzle, using compressible
flow theory.

Having now defined the boundary conditions within which the system
must perform, the specific intent, i.e. - what is the nozzle to
do, will be defined.

Considering that the process definition is "Ion Cluster Beam" the

primary goal of the source should be to generate clusters of the evaporant material; and if possible, not just random clusters but clusters of some controlled size and size distribution with ideally no monatomic species present.

The second goal of the source, which applies only to machines of the type used for this project, i.e. - where the source to substrate separation is approximately 24 inches, is to generate a fairly coherent beam. This will allow the flux on the substrate surface to be maintained at some reasonably high level as opposed to the low levels that would be obtained if the beam were allowed to fan out over a large area.

The next step in this process was to examine homogeneous and heterogeneous nucleation theories to determine which would more closely approximate the current configuration. This investigation made it apparent that trying to create a system which would operate in the homogeneous regime would be very difficult, if not impossible, since this type of nucleation requires a completely homogeneous system of extreme purity, i.e. - a system which contains no ions, contaminants, boundary surfaces or other elements that can assist in promoting the system over the activation energy barrier[10]. In addition, the supersaturation ratios required to generate significant numbers of clusters of any appreciable size, say greater than 200 atoms per, could have been difficult to obtain and control.

This leads us to try and establish a system that will operate

almost entirely in the heterogeneous regime. To accomplish this requires a means of producing intimate contact between the atoms in the expanding vapor and at the same time providing substantial contact to a free surface, such as the walls of the nozzle. The ideal candidate for this is the converging-diverging or deLeval nozzle which is shown in figure 26.

With this nozzle, throat exit velocities greater than Mach 1 can be obtained. When this situation exists, the streamlines intersecting the contoured sidewalls of the nozzle create Mach waves which are reflected inward causing areas of high compression or high supersaturation ratios and, as such, intimate contact of the atoms in the expanding vapor. In addition, the atoms contacting the sidewalls are assumed to be undergoing heterogeneous nucleation. By continually contacting the initial small clusters of atoms with the nozzle sidewalls and also with the areas of high compression created by the Mach waves, it is believed that a high percentage of the clusters reach the critical embryo size and, as such, can proceed to grow spontaneously into much larger clusters. Finally, by controlling the contour in the exit of the nozzle, a uniform flow at the exit can be produced.

B. Source Design

In redesigning the sources there were several aspects to consider. The first being to solve the existing problems of electric field cross talk, high vapor pressure in the vicinity of the ionizers,

BASIC DELEVAL NOZZLE CONFIGURATION

Supersonic beam consisting of single as well as clustered atoms.

Expansion/
Nucleation
Region

Vena
Contracta

vapor
flow

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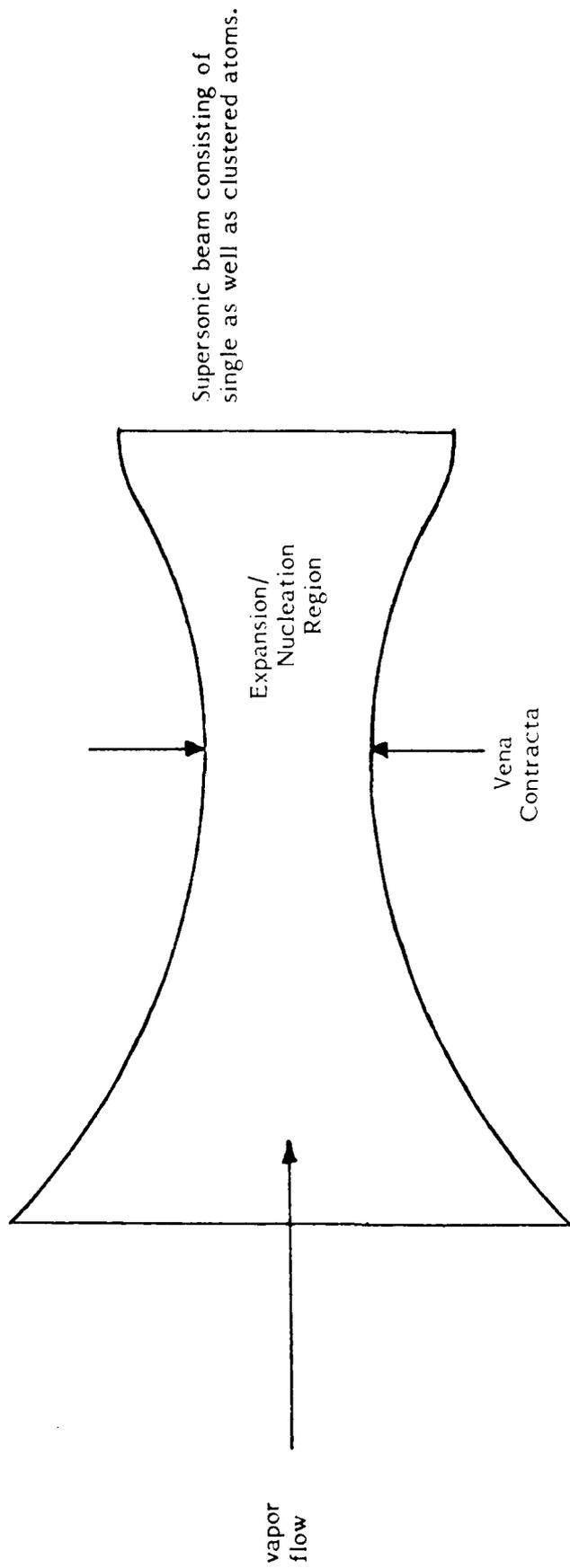
Figure 26

and poor ionizer efficiency as relates to the amount of current that could be extracted from the ionizer filaments and not necessarily the amount of ionization in the beam, although this was a prime consideration also. Secondly, the new source should be designed to incorporate the most recent improvements in ICB source design technology. These include uniform ionization of the beam with uniform ion distribution at the growing surface, as well as increased ion content within the beam.

To solve the first of the problems, a reference electrode, at ground potential, was incorporated into each source. In addition, a wire mesh, again at ground potential, was designed so as to completely surround the source in a cylindrical fashion. These two improvements should prevent any field cross talk by shunting any stray fields to ground.

The high vapor pressure in the vicinity of the ionizers was addressed by designing the new ionizers with cooling water jackets so that any stray portions of the beam would have a cold surface upon which to condense.

The final problem of poor ionizer efficiency was determined to be due to space charge effects which limited the current that could be extracted. To fix this problem, the new ionizers were designed with slightly larger and more open geometries so as to greatly reduce space charging effects and allow the ionizers to be operated in an emission limited mode.

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Having established the basic concept of source design necessary
to solve the existing problems, the task became one of
incorporating the latest improvements in source design.

The first improvement was to design an ionizer that would provide
uniform ionization of the cluster beam. To do this a modified
version of that used by Yamada[11], et. al. was employed. In
addition to this, it became apparent, through the work of Yamada,
et. al.,[12] that the uniformity of the ion distribution within the
beam was something that necessitated control. To accomplish
this, a set of electrostatic lenses was designed into each source.

Finally, in order to produce a source that could generate beams
with high ion content, as compared to current designs, the concept
of Mei and Lu[13] was incorporated into the design.

V. RESULTS AND DISCUSSION

Since the results of Phase I seemed to indicate that the problem with epi films grown by the ICB technique was crystallographic in nature, it was therefore decided that the characterization efforts would be concentrated in this area until a crystallographically "good" film was produced. From this point forward the characterization efforts would center around electrical and optical properties.

The Phase II results compiled in appendix A and summarized in Table IV. on the following pages, reveal some interesting aspects of the ICB films.

For instance, the first series of samples, Nos. 002 through 008, tend to indicate that below some critical temperature, apparently between 300 °C and 375 °C, the growth of large single crystal areas is not possible using just simple evaporation. Also, from the TEM micrographs it appears that the dislocations found in the samples grown at 375 °C, originate from surface contamination remaining on the substrate at the initiation of growth.

This is supported in part by the high resolution TEM micrographs of samples two and three. These indicate that in areas where the substrate is atomically "clean", growth tends to proceed naturally in an epitaxial fashion, but in areas where contamination exists, the growth becomes highly disoriented.

It could be speculated that the lack of an atomically clean substrate surface is due to inadequate substrate heating, i.e. - the substrate

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TABLE IV

S U M M A R Y

ICB CHARACTERIZATION RESULTS

Run No.	Film Morphology and Character	Channeling Pattern*	Structural Defects (cm^{-2})		Film Composition Atomic Percent							
			Stacking Faults	Others	Surface				Bulk			
					Ga	As	O	C	Ga	As	O	C
1	ABORTED											
2	Very smooth surface ----- Single crystal	Excellent	8E8	3E9	26	26	14	34	51	49	ND	ND
3	Smooth surface with unidirectional oval defects ----- Single crystal	Excellent		2E9	33	31	19	17	49	51	ND	ND
4	Smooth surface with numerous pits ----- Single crystal	Very diffuse	7E8	1E10	29	29	13	29	50	50	ND	ND
5	Smooth surface ----- Polycrystalline	No pattern			28	28	15	29	50	50	ND	ND
6	Very smooth surface ----- Polycrystalline	No pattern			21	23	15	41	48	52	ND	ND
7	Smooth surface ----- Polycrystalline	No pattern			24	23	18	36	50	50	ND	ND
8	Slightly rough surface ----- Polycrystalline	No pattern			34	34	14	18	50	50	ND	ND
9	ABORTED											

*Due to the SEM spot size, the channeling patterns may reflect all or part of the substrate, thus indicating higher film quality than is actually present on samples with rough partially coalesced films.

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TABLE IV

S U M M A R Y

ICB CHARACTERIZATION RESULTS

Run No.	Film Morphology and Character	Channeling Pattern*	Structural Defects (cm⁻²)		Film Composition Atomic Percent							
			Stacking Faults	Others	Surface				Bulk			
					Ga	As	O	C	Ga	As	O	C
10	Very rough surface ----- Partially coalesced single crystal islands	Diffuse	8E8	3E9	-	-	-	-	53	47	-	-
11	Very rough surface ----- Large grained polycrystalline	Extremely Diffuse			-	-	-	-	NA	NA	-	-
12	Rough surface ----- Single crystal	Slightly Diffuse			-	-	-	-	51	49	-	-
13	Very rough surface ----- Single crystal	Slightly Diffuse	4E8	2E9	-	-	-	-	51	49	-	-
14	Very rough surface ----- Single crystal	Slightly Diffuse		2E9	-	-	-	-	50	50	-	-
15	Very rough surface ----- Partially coalesced single crystal islands	Slightly Diffuse		2E9	-	-	-	-	50	50	-	-
16	Very rough surface ----- Partially coalesced single crystal islands	Slightly Diffuse	4E8	9E9	-	-	-	-	50	50	-	-
17	Very rough surface ----- Small grained polycrystalline	Diffuse		5E10	-	-	-	-	50	50	-	-
18	Very rough surface ----- Uncoalesced polycrystalline islands	Slightly Diffuse		2E10	-	-	-	-	50	50	-	-

*Due to the SEM spot size, the channeling patterns may reflect all or part of the substrate, thus indicating higher film quality than is actually present on samples with rough partially coalesced films.

TABLE IV

S U M M A R Y

ICB CHARACTERIZATION RESULTS

Run No.	Film Morphology and Character	Channeling Pattern*	Structural Defects (cm^{-2})		Film Composition Atomic Percent							
			Stacking Faults	Others	Surface				Bulk			
					Ga	As	O	C	Ga	As	O	C
19	Very rough surface ---- Partially coalesced polycrystalline islands	Slightly Diffuse		1E10	-	-	-	-	45	55	-	-
20	Very rough surface ---- Polycrystalline	Extremely Diffuse			-	-	-	-	50	50	-	-
21	Rough surface ---- Polycrystalline	Extremely Diffuse			-	-	-	-	52	48	-	-
22	Rough surface ---- Polycrystalline	No pattern			-	-	-	-	50	50	-	-
23	Rough surface ---- Partially coalesced polycrystalline islands	Extremely Diffuse			-	-	-	-	53	47	-	-
24	Very rough surface ---- Partially coalesced polycrystalline islands	Excellent		1E10	-	-	-	-	50	50	-	-

*Due to the SEM spot size, the channeling patterns may reflect all or part of the substrate, thus indicating higher film quality than is actually present on samples with rough partially coalesced films.

temperature never went above 375 °C. Under these conditions it is reasonable to assume that a large portion of any arsenic oxides were desorbed from the surface but any gallium oxides were not since studies have shown that the gallium oxides do not become volatile until the temperature is well above 500 °C.

This problem should be solvable in the ICB system by exposing the substrate surface to a beam of arsenic cluster ions or some inert gas ions prior to initiation of growth. Unfortunately, due to the mechanical and design problems previously discussed it was not possible to implement effectively this procedure.

Another important piece of data is the electron channeling patterns. Notice that the pattern quality on samples two and three is excellent and on several other samples the pattern is only slightly diffuse.

If one were to consider only this data it would appear that the film was of high crystallographic quality, but upon examining the TEM micrographs from these samples, it quickly becomes obvious that the films are composed of large grains.

Taking this data into consideration, a brief re-evaluation of the Phase I results is in order since, during Phase I the methods of structural characterization consisted of x-ray diffraction curves and electron channeling patterns. Using only these techniques, the problem outlined above possibly existed, i.e. - these techniques indicated structurally sound material but both use relatively small spot sizes and possibly would not have revealed the presence of very large grains.

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It is the boundaries created by these large grains that would easily explain the lack of mobility that was seen in the Phase I samples and not the postulated point or anti-site defects although these may be of importance in producing the ultimate film quality.

This now makes the Phase I data much more self consistent.

Finally since the balance of the Phase II runs were made using a columnating nozzle of some type and considering the misalignment of the source centerline with the substrate, the results of these runs most closely resemble simple evaporation at extremely low fluxes of gallium and arsenic.

This extremely slow growth rate coupled with the system inability to do in-situ cleaning or reasonable beam ionization is most likely the reason for the very large numbers of uncoalesced and partially coalesced islands observed.

VI. CONCLUSION

Although the data collected to this point from both phases of the project is somewhat inconclusive, it should be noted that the process as conceived, i.e. - operation with ionization and acceleration of clustered particles, has not been realized. This has resulted in a process consisting of ion assisted deposition, but not ionized cluster deposition, or simple evaporation, as is the case for Phase II.

Since in both phases, the inability to implement the process, as conceived, was the result of a multitude of mechanical and design problems, it became very apparent that the optimization of the hardware is as important as any other factor to the success of the process.

For these reasons, the process of "Ion Cluster Beam Deposition" as relates to gallium arsenide has not as yet received an appropriate evaluation.

Therefore, it is believed that with the redesign effort currently underway, the mechanical and design problems will be rectified to the point where a true evaluation of the ICB process can be made. Also, considering some of the current results, it is felt that a viable process can be generated and with the proper hardware in place, a process for growth of gallium arsenide at or below 400 °C is still a possibility.

VII.   ACKNOWLEDGEMENTS

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